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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

895. **The changing aspect of chemical analysis.** H. N. Wilson (I.C.I. Ltd., Billingham, Co. Durham, England). *Analyst*, 1960, **85**, 540–550.—A lecture.

896. **Conference on automatic chemical analysis. New York, Nov. 1959.** *Ann. New York Acad. Sci.*, 1960, **87**, 611–951.—The following papers are of analytical interest. **Automatic chemical analysis,** R. H. Muller, 611–615. **Automation of enzyme determinations: phosphohexose isomerase,** M. K. Schwartz, G. Kessler and O. Bodansky, 616–628. **Cholinesterase-activity determination in an automated analysis system,** G. D. Winter, 629–635. **Continuous automatic integrated flame photometry,** J. Isreeli, M. Pelavin and G. Kessler, 636–649. **Determination of carbon dioxide in blood serum,** L. T. Skeggs, jun., 650–657. **Continuous determination *in vivo* of blood glucose in human subjects,** C. Weller, M. Linder, A. Macaulay, A. Ferrari and G. Kessler, 658–668. **Random selection system for dynamic biochemical analysis,** R. Jonnard, 669–728. **Continuous automatic chemical analysis *in vivo*,** A. Ferrari, G. Kessler, F. M. Russo-Alesi, J. M. Kelly, C. Vanderwende and L. E. van Petten, 729–744. **Automatic analysis of amino-acids,** K. R. Woods and R. L. Engle, jun., 764–774. **Solution of problems involving special handling of samples and reagents in an automatic system,** I. E. Taylor and M. M. Marsh, 775–781. **Automation of the microbiological assay of antibiotics with an auto-analyser instrumental system,** J. R. Gerke, T. A. Haney, J. F. Pagano and A. Ferrari, 782–791. **Nitrogen determination by a continuous digestion and analysis system,** A. Ferrari, 792–800. **Methods for the determination of ammonia in biological materials on the auto-analyser,** E. E. Logsdon, 801–807. **Continuous automatic chemical analysis of nitrate in the presence of ammonia and urea,** E. W. Catanzaro, 803–812. **Simultaneous determination of sample and blank by means of dual and differential colorimetry in the continuous analysis of penicillin in fermentation media,** F. M. Russo-Alesi, C. Sherman, J. M. Kelly and A. Ferrari, 822–829. **Partial or complete automation as alternatives available to the analyst,** D. A. Patient, 830–843. **Recent developments in automatic colorimetric chemical analysis instruments,** R. T. Sheen and E. J. Serfass, 844–856. **Automated method for determination of terramycin,** T. C. Grenfell, D. J. McLaughlin and J. M. Kelly, 857–863. **Continuous and automatic analysis of process streams by gas chromatography,** H. J. Maier and H. N. Claudy, 864–874. **Automated enzymatic analysis for L-lysine via decarboxylation,** G. E. Schaiberger and A. Ferrari, 890–893. **Automation of sugar analysis in barometric condensers and boiler waters,** E. H. Baum, 894–903. **Method development for phosphate analysis with the auto-analyser,** D. P. Lundgren, 904–910. **Automatic analytical instrumentation in the modern power plant,** W. A. Crandall, 911–923. **Continuous sampling and automatic analysis for silica in modern high-pressure boilers,** K. F. Schunk, 924–933. **Design of automatic trace analysers,** H. J. Noebels, 934–943. **Problems on automation of chemical analysis and industrial process,** J. Kelly, 944–951.

897 **Kinetic methods in chemical analysis.** K. B. Yatsimirskiĭ (Chem. Technol. Inst., Ivanovo, USSR). *Chem. Listy*, 1960, **54** (8), 795–805.—A review with 106 references. J. ZÝKA

898. **Colloquium Spectroscopicum Internationale** (Luzern, Sept. 1959). Proceedings publ. by H. R. Sauerländer & Co., Aarau (Switzerland). 319 pp.—The following are the titles of papers of analytical interest. **Quantitative flame spectral analysis,** R. Mavrodineanu, 15–29 (in French). **Problems of mass spectroscopy,** E. Schumacher, 29–39 (in German). **Microwave spectroscopy,** H. H. Günthard, 39–44 (in English). **The relation of the spectral character of a spark discharge to its current - time curve,** K. Laqua and W. D. Hagenah, 45–49 (in German). **The action of the carrier in vaporisation of trace impurities in spectral analysis,** L. Pszonicki, 50–54 (in German). **Performance and proof in spectrographic trace analysis,** A. Schöntag, J. Roth and M. Lechner, 58–62 (in German). **The influence of electron pressure in trace analysis by the luminous arc,** V. M. Vukanović, 62–66 (in German). **The volatilisation of metallic electrodes in the spark,** G. Richter, 66–69 (in German). **Emission spectroscopy of radioactive elements,** J. G. Conway, 70–73 (in English). **Spectroscopic properties of photographic emulsions,** A. Arrak, 73–76 (in English). **Comparative observations on the limits of detection in photographic and photo-electric spectrochemical analysis,** H. de Laffolie, 76–81 (in German). **The convertibility of spectrochemical procedures with complete volatilisation,** H. J. Eichhoff and N. W. H. Addink, 89–92 (in German). **Application of a universal spectrographic microanalytical procedure to mineralogical and metallographic problems,** J. Niebuhr and C. Pothmann, 93–96 (in German). **Arc spectrography with controlled emission energy in argon for the overall determination of tracers,** Z. Hainski and P. Herman, 97–104 (in French). **The influence of the metallic state on the spectral emission of very high strength steels,** A. Camuñas and M. V. de la Peña, 105–107 (in French). **Spectrographic control analysis in the production of pure hafnium and zirconium,** W. J. Price, 107–112 (in English). **Analysis by emission spectrography in the far ultraviolet of powders containing germanium, arsenic, selenium, bromine, tin, antimony or tellurium,** G. Balloffet, J. Romand and B. Vodar, 112–114 (in French). **Different influences of the sifter-electrode procedure in spectral analysis,** J. Czakow, 114–118 (in German). **Residual arc spectra of seventy elements diluted in copper,** C. H. Corliss, W. F. Meggers

and B. F. Scribner, 119–121 (in English). **Study of the electrode processes occurring in the spectrochemical analysis of uranium oxides,** S. Marinković, 121–126 (in English). **Determination of cations in lubricating oils,** J. M. Lopez de Azcona and J. Sanz de la Rosa, 129–130 (in French). **Determination of phosphorus, zinc, calcium and barium in new oils by direct-reading emission spectrography,** G. de Clippeleir, G. Souillard and J. van Rysselberge, 130–140 (in French). **An original technique for spectrographic control of thin plates,** M. Humbert, 140–145 (in French). **Emission spectrographic trace analysis with the double-arc method,** E. Schroll, M. Brandenstein, I. Janda and W. Rockenbauer, 145–149 (in German). **Reciprocal effects of alloy constituents in the excitation of brasses and analogous effects between substances simultaneously excited in a flame,** J. van Calker, 150–155 (in German). **A new method of spectrographic analysis by injection of solutions by means of a gas,** I. A. Voinovitch, N. Liodec, J. Debras-Guédon and J. Vilnat, 155–159 (in French). **Determination of oxygen and nitrogen in titanium samples with a condensed spark in a vacuum,** J. Romand, G. Balloffet and B. Vodar, 160–162 (in French). **The influence of ethylenediaminetetra-acetic acid on the emission errors of calcium and strontium in the flame,** J. Malinowski, 167–171 (in German). **New direct method of analysis of Sr, Ca, Na, Li and K in the presence of Al, Ti, Fe and Mg in silicates by flame spectrophotometry,** J. Debras-Guédon and I. A. Voinovitch, 171–175 (in French). **Flame-photometric determination of traces of sodium in aluminium,** K. H. Neeb, 176–177 (in German). **Flame-spectrophotometric determination of lithium in nuclear materials,** J. Perman, 177–184 (in German). **Determination of rapidly fluctuating oxygen content in gas mixtures,** L. Krauss and H. Prugger, 188–191 (in German). **Use of the "copper spark" method for the determination of F, Cl, Br and I in solutions,** J. Fijałkowski and O. Szczerbińska, 191–195 (in French). **Use of the tungsten electrode in routine spectral analysis in the control of manufacture,** V. Mathien, M. Lacomble and L. Charlet, 195–198 (in French). **Application of the Noar cellulose pellet arc method to the Quantometer,** A. Kvalheim and K. S. Vestre, 198–204 (in English). **New methods of photo-electric measurement in emission spectral analysis,** G. Cannet, J. Pomey and C. Pournin, 219–233 (in French). **The use of emission- and mass-spectroscopy in the analysis of high-purity metals,** F. Rohner, 249–251 (in German). **Analysis of traces of oxygen in carbon dioxide by mass spectrometry,** G. Nief and M. Séverin, 255–258 (in French). **Secondary ion-processes in mass spectrometry,** A. Quayle, 259–267 (in English). **Mass spectra of simple linear and cyclic silicon-methylene compounds,** F. Aulinger, 267–271 (in German). **Comparison of X-ray fluorescence and emission spectroscopy for analysis,** J. R. Stansfield, 278–281 (in English). **X-ray emission spectrography in the metal industry,** C. M. Davis, 281–285 (in English). **The use of X-ray quantometry in the steelworks,** R. Loude and R. Billet, 286–290 (in French). **Rapid analysis of metallurgical products by X-ray fluorescence,** A. Hannick, A. Hans, L. Flechet and Y. Houbart, 290–298 (in French). **Application of X-ray fluorescence spectroscopy in the field of light metals,** H. Pfundt, 299–303 (in German). **Experiences with an X-ray fluorescence vacuum spectrograph,** J. L. de Vries, 303–306 (in English). **Applications of β-ray fluorescence to the measurement of thickness of deposits and to analysis,** P. Martinelli and G. Seibel, 307–313 (in French).

899. **Recent trends in polarography.** J. Heyrovský (Polarographic Inst., Czech. Acad. Sci., Prague). *Angew. Chem.*, 1960, **72** (13), 427–432 (in German). —A report of the Nobel Lecture delivered on December 11, 1959.

900. **Development in the polarographic analysis of inorganic ions.** I. V. Pyatnitskiĭ. *Zavod. Lab.*, 1960, **26** (7), 798–810.—A review with 227 references is presented. G. S. SMITH

901. **Some statistical considerations.** A. R. Bloemena (Math. Centrum, Amsterdam, Netherlands). *Chem. Weekbl.*, 1960, **56** (32), 465–471.—Some statistical procedures are described which may be used when considering the accuracy of analytical methods, *e.g.*, determination of the confidence interval, the *t*-test and the *F*-test. Difficulties in the interpretation of duplicate observations, obtained by carrying out two determinations simultaneously, are discussed. M. J. MAURICE

902. **Precision and accuracy of analytical methods.** M. J. Maurice (Centr. Anal. Lab., N.V.A.K.U., Arnhem, Netherlands). *Chem. Weekbl.*, 1960, **56** (33), 478–485.—A review is given of some methods for establishing the precision and the accuracy of analytical methods, whereby use is made of some statistical tests. The variance is used in reporting the precision. Two cases are distinguished, *viz*, the variance is constant at varied amounts of the compound to be determined and the variance increases with increasing amounts of the compound. Examples of both are given. Further, the calculation of the variance of a test method requiring a blank determination is discussed. There are essentially two ways in which the accuracy of an analytical method can be established, *viz*, by comparison of the results obtained with the known content of a standard preparation and by comparison of test results with those obtained by one or more independent methods of known accuracy. In both cases the *t*-test is used. Some examples are given as illustrations. (16 references.)
M. J. MAURICE

903. **The tracing of sources of error in analytical chemistry.** J. Agterdenbos (Anal. Chem. Lab., Rijksuniv., Utrecht, Netherlands). *Chem. Weekbl.*, 1960, **56** (34), 489–494.—In chemical analysis two kinds of error may occur, *viz*, systematic errors and errors due to chance alone. Both kinds of error may be dependent or independent of the amounts to be determined; in the first case they are often proportional to these amounts. It is shown that, by application of statistical methods to the results found, the errors can be classified, so that sometimes the source of these errors can be eliminated. Examples are given to illustrate this. The influence of errors made before the extinction measurement in spectrophotometry, in the extinction range where the dispersion is minimum, is discussed. The chemical interferences that arise during preparation of the coloured soln. are classified into errors that are constant at different concn. and into errors that are proportional to the amounts determined.
M. J. MAURICE

904. **Glass in the laboratory.** R. W. Douglas (Univ., Sheffield, England). *Lab. Practice*, 1960, **9** (8), 585–590.—A lecture.

905. **Nucleation in precipitation reactions from homogeneous solution.** R. B. Fischer (Gates and Crellin Lab., California Inst. of Technol., Pasadena,

U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1127–1130.—In the method described, a sample suspension is mixed with a standard suspension, and the particles formed are counted in various areas under a microscope. In pptn. from homogeneous soln., nucleation occurs early in the reaction. K. A. Proctor

906. **Preparation of spectroscopically pure cyclohexane.** N. C. Saha and N. G. Basak (Central Fuel Res. Inst., Jealgora, India). *J. Sci. Ind. Res., India, B*, 1960, **19** (6), 227.—Cyclohexane containing less than 0·05 mg of benzene per ml can be purified for spectroscopic use by passage through a glass column (3 cm × 50 cm) packed with dry silica gel (100 to 200 B.S. mesh) activated by heating for 1 hr. at 350°. Passage is made in an atmosphere of N at a pressure up to 381 torr. Ten litres can be passed through such a column before a 'break point' appears. For material containing more than 0·05 mg of benzene per ml, a chemical method is recommended. The sample is shaken for 30 min. in a separating-funnel with one-tenth of its vol. of 10% oleum. After separating the browned oleum, the cyclohexane is washed several times with 5% oleum till the oleum ceases to become coloured, then with an equal vol. of H_2O, followed by an equal vol. of 2% aq. alkali, and finally with H_2O until free from alkali. After drying overnight over anhyd. $CaCl_2$, the cyclohexane is distilled in glass apparatus and, if necessary, passed through a silica gel column as described above. Cyclohexane so treated shows no absorption at 255 to 300 mμ. J. I. M. Jones

907. **Indicators.** R. Belcher (Univ., Birmingham, England). *Lab. Practice*, 1960, **9** (8), 568–570.—A lecture.

908. **Azo-dyes as indicators for calcium and magnesium.** H. Diehl and J. Ellingboe (Iowa State Univ., Ames, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1120–1123.—An examination of 26 mono-azo-compounds shows that, for union with Ca^{2+} or Mg^{2+}, such a dye must contain two hydroxyl groups in *o*- and *o'*-positions, respectively, to the azo-group or one *o*-hydroxyl group and one *o'*-carboxyl group in the molecule. In aq. soln. the reaction with the metal ion always forms compounds of 1 : 1 ratio. The formation constant for the magnesium compound is always greater than that for the corresponding calcium compound, whilst the constant for the *o*-hydroxy-*o'*-carboxyazo-metallo-compound is approx. one-hundredth of that for the *oo'*-dihydroxyazo-compound. (33 references.) W. J. Baker

909. **Indicator for the titration of calcium plus magnesium with ethylenedinitrilotetra-acetate.** F. Lindstrom and H. Diehl (Clemson Coll., S.C., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1123–1127.—The recommended new indicator is 1-(2-hydroxy-5-methylphenylazo)-2-naphthol-4-sulphonic acid and has been given the common name Calmagite. The preparation of this dye is described. It is preferred to Eriochrome black T because the colour change at pH 10, though the same, is much sharper and clearer, whilst its aq. soln. is stable indefinitely. It can be substituted for the older indicator without any change in procedure, the same masking agents for Cu, Fe and Al being equally effective. High concn. of Na^+ interfere, so that any neutralisation before the EDTA titration should be effected with KOH. W. J. Baker

910. **Use of metal fluorechromic indicator for the complexometric titration of metal ions.** M. R. Verma and K. C. Agarwal (Nat. Phys. Lab., New Delhi, India). *J. Sci. Ind. Res., India, B*, 1960, **19** (8), 319–320.—Tervalent Cr, alone or in the presence of dichromate and Fe^{3+}, is determined by titration with the disodium salt of Chenta acid (diaminocyclohexanetetra-acetic acid) (**I**) and Th by titration with **I** or EDTA (**II**), with calcein as indicator on a semi-micro scale. *Procedure for Cr^{3+}*—Boil the soln. with an excess of **I** at pH 4 for 30 min., cool, and adjust to pH 4·6 with Na acetate - acetic acid buffer. Add 1 drop of calcein soln. (2%) and titrate with standard $CuSO_4$ soln. under an u.v. lamp with a Wood's filter till the fluorescence is discharged. *Procedure for Fe^{3+}*—Boil the soln. with an excess of **I** at pH 3·5 to 4·0 for 10 min., cool, buffer to pH 4·6 and titrate as described for Cr^{3+}. *Procedure for Th*—Add an excess of **I** or **II**, buffer to pH 4·6 and titrate with $CuSO_4$ as described for Cr^{3+}. J. W. Price

911. **Haematoxylin as indicator in the chelatometric titration of metals.** A. C. S. Costa (Univ. Bahia, Salvador, Brazil). *Anal. Chim. Acta*, 1960, **23** (2), 127–130 (in English).—The use of haematoxylin as an indicator in the titration of Bi, Zr, Pb, Cu, Cd, Co and Ni with EDTA has been investigated. Excellent results were obtained for Bi at pH 1 to 2, even in the presence of other metals. Zirconium was satisfactorily titrated at pH 1 to 1·5 with haematoxylin screened with methylene blue. Several anions and cations interfered. None of the other metals was titrated satisfactorily. G. S. Roberts

912. **Pyromellitein indicators. III. Lake formation.** J. A. Bishop (Coll. of Engng, Newark, N.J., U.S.A.). *Anal. Chim. Acta*, 1960, **23** (2), 124–126 (in English).—Indicators made by condensing pyromellitic acid dianhydride with phenols (*cf. Anal. Abstr.*, 1960, **7**, 4096, 4623) have been found to form coloured lakes with some metal hydroxides. A few give intense colours with < 200 p.p.m. of the metal ion and may find use in micro-analysis. G. S. Roberts

913. **Recent progress in gas-phase partition chromatography.** P. Chovin (Lab. Municipal de Paris, France). *Bull. Soc. Chim. France*, 1960, (5), 755–768.—Recent developments in nomenclature, theory, columns and detectors are reviewed. The use of traps and pre-columns and preparative columns is discussed. Continuous and circular gas chromatography are described. (96 references.) E. J. H. Birch

914. **Ion-exchange techniques.** J. E. Salmon (Battersea Coll. of Technol., London, England). *Lab. Practice*, 1960, **9** (8), 571–576.—A lecture.

915. **Recent developments in colorimetry.** B. Trémillon (École de Phys. et de Chim., 10 rue Vauquelin, Paris, France). *Bull. Soc. Chim. France*, 1960, (5), 775–782.—The theory of colorimetric determinations is reviewed and differential colorimetry is discussed. The application of colorimetric methods to the determination of traces, and the precision of the measurements, the elimination of interfering substances, the determination of substances by the decrease in extinction in their presence, and various titrimetric methods (including the coulometric production of the reagents in the spectrophotometer cell) are discussed. (88 references.) E. J. H. Birch

916. **Least-squares treatment of spectrometric data.** H. A. Barnett and A. Bartoli (U.S. Steel Appl. Res. Lab., Monroeville, Pa.). *Anal. Chem.*, 1960, **32** (9), 1153–1156.—The treatment described yields equations for calculating the concn. of each of *n* components in a mixture from extinctions measured at *n* selected wavelengths. The method is applicable to mass-spectrometric data from the analysis of light-oil fractions. K. A. PROCTOR

917. **The electron probe. An added dimension in chemical analysis.** L. S. Birks (U.S. Naval Res. Lab., Washington, D.C.). *Anal. Chem.*, 1960, **32** (9), 19A–28A.—By means of the electron probe, quant. analyses of all elements above at. no. 11 can be carried out on <1 $\mu\mu$g of material in any selected 1 square-micron area on a specimen surface. A focused beam of 10 to 50-keV electrons is used to excite characteristic X-ray spectra, and standard X-ray optics are used to measure the spectral intensity. Applications discussed include segregations in alloys and minerals, diffusion or corrosion zones, and thin surface layers. K. A. PROCTOR

See also Abstract—**1080**, Tetraphenylstibonium sulphate as reagent for org. acids.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

918. **Determination of complexed solvent molecules of extracted inorganic compounds.** E. Jackwerth and H. Specker (Inst. f. Spektrochem. und angew. Spektroskop., Dortmund-Aplerbeck, Germany). *Z. anal. Chem.*, 1960, **176** (2), 81–87 (in German).—The procedure of continuous concentration variation (*cf. Anal. Abstr.*, 1960, **7**, 1251) is applied to the determination of the solvation number of extracted inorganic compounds in organic solvents, and examples are given.

B. B. BAUMINGER

919. **Paper-chromatographic analysis of acids (horizontal migration method). VII.** V. K. Mohan Rao (Central Drug Res. Inst., Lucknow, India). *J. Sci. Ind. Res., India, B*, 1960, **19** (7), 265–267.—The R_F values of F^-, Cl^-, Br^-, I^-, ClO_3^-, BrO_3^-, IO_3^-, SCN^-, NO_3^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, SO_4^{2-}, CrO_4^{2-} and AsO_4^{3-} are determined by circular paper chromatography (Rutter's method) by the use of the org. layers of n-butanol satd. with 1·5N-aq. NH_3; n-butanol - pyridine - water - aq. NH_3 (*d* 0·96) (80:40:77:8); 2,6-lutidine - water (7:3); collidine satd. with water; n-propanol - aq. NH_3 (*d* 0·88) (4:1) or 95% ethanol - aq. NH_3 (*d* 0·88) - water (20:1:4). The effect of variation of pH on the R_F of the halides is insignificant. The presence of NH_4^+ or Li^+ increases the R_F of the halide ion compared with that obtained when the cation is Na^+ or K^+. E. J. H. BIRCH

920. **Paper-chromatographic separation of silver, lead and mercurous mercury.** V. K. Mohan Rao (Central Drug Res. Inst., Lucknow, India). *J. Sci. Ind. Res., India, B*, 1960, **19** (5), 171–173.—Silver, Pb and Hg^I can be separated by ascending paper chromatography on Whatman No. 1 paper with 0·2N-H_2SO_4 or aq. NH_3 - NH_4NO_3 soln. as mobile phase. The metals are applied to the paper as the nitrates, and located after separation by exposing the paper first to NH_3 and then to H_2S. Lead (1 to 8 μg) and Ag (1 to 10 μg) can be determined by developing the unknown with 0·3N-aq. NH_3 as mobile phase and making a visual comparison with standard chromatograms. W. T. CARTER

921. **Chromatographic behaviour of cations. II. Separation of platinum, palladium, gold and copper.** T. H. V. Setty (Central Coll., Bangalore, India). *Current Sci.*, 1960, **29** (6), 221–222.—Solutions of the chlorides of the metals named ($\simeq$0·01M) were examined by the circular paper-chromatographic technique previously described (*Ibid.*, 1956, **25**, 218), with various primary alcohols containing various concn. of HCl as solvents. For the same mixture of alcohol - HCl - water (17:2:1), the R_F values decreased with increase in the chain-length of the alcohol, while for n-butanol saturated with various concn. of HCl (0·1 to 4N), the R_F values, the sharpness of the boundaries and the visible intensities of the bands increased with increase in the acid concn. Good separations were achieved with benzyl alcohol - conc. HCl - water (50:5:3); the R_F values for Cu, Pd and Pt were 0·38, 0·52 and 0·89, respectively, while Au was extracted with the alcohol phase, which travels ahead of the water front. R. E. E.

922. **Chromatographic separation of inorganic anions on specially prepared calcium sulphate sticks.** B. N. Sen (Syamsundar Coll., West Bengal, India). *Anal. Chim. Acta*, 1960, **23** (2), 152–153 (in English).—Sticks composed of $CaSO_4.2H_2O$ (45%) and plaster of Paris (55%) prepared by the technique described by Sen (*Anal. Abstr.*, 1955, **2**, 1447) were successfully used to separate PO_4^{3-} from HPO_3^{2-}, AsO_4^{3-} from AsO_3^{3-}, AsO_4^{3-} from HPO_3^{2-}, AsO_3^{3-} from HPO_3^{2-}, $S_2O_3^{2-}$ from SCN^-, $[Fe(CN)_6]^{4-}$ from SCN^-, $[Fe(CN)_6]^{3-}$ from SCN^- and $[Fe(CN)_6]^{4-}$ from $[Fe(CN)_6]^{3-}$. G. S. ROBERTS

923. **An ion-exchange selectivity scale of cations based on equilibrium distribution coefficients.** F. W. E. Strelow (Nat. Chem. Res. Lab., S. African C.S.I.R., Pretoria). *Anal. Chem.*, 1960, **32** (9), 1185–1188.—The results of a study of distribution coefficients of cations in HCl are given as well as the results of an investigation into the relationship between distribution coefficients and elution curves. The distribution coefficients of 43 cations in HCl, with the cation-exchange sulphonated polystyrene AG 50W-X8, are enumerated. K. A. PROCTOR

924. **Determination of hydrogen in metals: a carrier gas method.** F. R. Coe (Brit. Welding Res. Ass., Abington, Cambridgeshire). *Research*, 1960, **13** (8), 323–327.—The method described combines an inert carrier technique with thermal-conductivity detection. It is flexible and suitable for routine control purposes. O. M. WHITTON

925. **New colour reaction for alkali-metal ions.** F. E. Critchfield and J. B. Johnson (Union Carbide Chem. Co., S. Charleston, W. Va., U.S.A.). *Talanta*, 1960, **5** (1), 58–61.—When a soln. of alkali-metal ions in acetic anhydride is heated with a soln. of citric acid in acetylacetone and toluene a violet - red colour with max. absorption at 560 mμ is developed. With K^+ and Na^+ the reaction is quant. and Beer's law is followed. Besides the alkali metals, only tertiary amines give a colour reaction, but

Fe^{3+}, water, alcohols and some acids interfere by inhibiting the development of the colour.

G. BURGER

926. **Chloranilic acid as a reagent in the paper chromatography of inorganic compounds. I. Alkali and alkaline-earth metals.** H. S. R. Barreto and R. C. R. Barreto (Escola Nac. Agron., Univ. Rural, Rio de Janeiro, Brazil). *J. Chromatography*, 1960, **4** (2), 153–155 (in English).—Chloranilic acid as a 0·1% soln. in ethyl ether is an extremely sensitive reagent for alkali and alkaline-earth metals. By using the dipping technique, 0·2 to 1 µg of Na, K and Li, and 0·5 to 5 µg of Ba, Ca, Sr and Mg can be detected. Alkali metals give violet spots at low concn., changing to brownish yellow at higher concn., but with alkaline-earth metals the colour varies from yellow to brownish yellow.

S. M. MARSH

927. **Flame-spectrophotometric determination of lithium in lithium minerals.** J. L. Kassner, V. M. Benson and E. E. Creitz (Bureau of Mines, U.S. Dept. of the Interior, University, Ala.). *Anal. Chem.*, 1960, **32** (9), 1151–1153.—The simple and rapid method described can be used to determine Li in minerals at concn. of 0·1 to 8·0%. The sample is decomposed with a mixture of HNO_3, HF and $HClO_4$, and the removal of interfering ions is not necessary, as the effects of these are largely eliminated by buffering the soln. within the pH range of 1 to 4 with a citric acid - ammonium citrate buffer. Under these conditions Be is the only element commonly found that causes any error. The precision and accuracy are stated to be good.

K. A. PROCTOR

928. **Determination of oxygen in sodium. The mercury method: its use in the case of very low concentrations.** L. Champeix, R. Darras and J. Duflo (Centre Études Nucl. Saclay, Gif-sur-Yvette, France). *J. Nuclear Materials*, 1959, **1**, 113–119 (in French).—The application of the mercury method (*cf.* Pepkowitz *et al.*, *Anal. Chem.*, 1950, **22**, 10) to micro amounts of O (<40 p.p.m.) was examined in a modified apparatus operating under vacuum. The NaOH formed was determined by flame spectrophotometry. As little as 7 p.p.m. was determined with a reproducibility of ± 1·5 p.p.m.

N. E.

929. **Adaptation of the bitartrate method for the estimation of potassium in sea bittern.** B. K. Shukla and D. J. Mehta (Central Salt Res. Inst., Bhavnagar, India). *Z. anal. Chem.*, 1960, **176** (5), 355–359 (in English).—The bitartrate method has been modified for the determination of K in the presence of $\not>$2 g of $MgCl_2$, 0·2 g of NaCl and 0·7 g of $MgSO_4$ in a 25-ml sample. *Procedure*—To the sample (25 ml) are added 15 ml of 0·38M-sodium hydrogen tartrate (20 ml in the presence of Mg salts) and 5 ml of 20% tartaric acid soln. The pptn. is completed by scratching the sides of the beaker for 5 min., followed by the dropwise addition of 65 ml of ethanol over a further period of 6 to 8 min. The difference between results by this method and those by the cobaltinitrite method, in the presence of $\not>$0·7 g of $MgSO_4$ per 25 ml, is < −1%.

E. G. CUMMINS

930. **Application of conversion X-ray spectra to isotopic analysis. Resolution of mixtures of caesium-134 and caesium-137.** M. T. Kinsley, J. B. Cumming and H. L. Finston (Brookhaven Nat. Lab., Upton, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1081–1083.—Mixtures of the isotopes are analysed by γ- and X-ray spectrum techniques. The γ-ray photopeak at 800 keV is used to determine ^{134}Cs. At 32 keV, the X-rays resulting from internal conversion electrons are measured and used to calculate the ^{137}Cs. The accuracy of the ^{134}Cs determination is within 1%. The ^{137}Cs content of mixtures having ^{134}Cs to ^{137}Cs activity ratios as high as 19 : 1 is determined with an accuracy better than 7%.

K. A. PROCTOR

931. **Metallic compounds of β-dioximes. I. The copper - acetylacetone dioxime complex: composition, properties and analytical aspects.** A. H. I. Ben-Bassat, Y. Sa'at and S. Sarel (School of Pharm., Hebrew Univ., Jerusalem, Israel). *Bull. Soc. Chim. France*, 1960, (5), 948–954.—The complex formed with Cu and acetylacetone dioxime is studied spectrophotometrically and potentiometrically and it is shown that the absorption max. is at 600 mµ, that the optimum pH for formation is 5·5, and that Beer's law is followed for concn. of Cu between 0·001 and 0·0025M. Job's and other methods show a 1 : 1 ratio of Cu to dioxime in the complex. The oxidation no. of Cu in the complex is shown to be +1. Colorimetric titration shows a break in the curve at a ratio of Cu to dioxime of 2 : 3 as well as at 1 : 1. The former point is considered to correspond to the required excess of dioxime for complete stabilisation and not to a definite compound. The reaction of the complex with NaOH is discussed. The instability const. of the complex is $1{\cdot}26 \times 10^{-9}$.

E. J. H. BIRCH

932. **Titrimetric determination of copper using diphenylthiovioluric acid.** R. P. Singh and N. R. Banerjee (Univ. Delhi, India). *Current Sci.*, 1960, **29** (6), 223–224.—The gravimetric method previously described (Singh and Shankar, *J. Indian Chem. Soc.*, 1955, **32**, 557) has been modified. The Cu (≃20 mg) is pptd. with the ammonium salt of diphenylthiovioluric acid (1,3-diphenyl-2-thioalloxan 5-oxime) (*loc. cit.*) and the ppt. is filtered off and washed with hot water. It is then dissolved in acetone, the soln. is evaporated to dryness and the residue is treated with ≃20 ml of 10% NaOH soln. and transferred to a stoppered flask. The soln. is then acidified with HCl and an equal vol. is added in excess; 0·1N-$KBrO_3$ (approx. 10 ml in excess) is added through a dropping-funnel, the acid concn. is adjusted to 3 to 4N, and the mixture is set aside for 15 min. A measured excess of 0·1N-As_2O_3, containing a little methyl orange, is added and the excess is back-titrated with 0·1N-$KBrO_3$; 1 ml ≡ 0·1821 mg of Cu. The error for 20 to 30 mg of Cu is ≃0·8%.

R. E. E.

933. **Determination of metal ions by means of TAA [thioacetamide] and EDTA. II. Determination of copper.** G. C. Krijn, C. J. J. Rouws and G. den Boef (Univ., Amsterdam, Netherlands). *Anal. Chim. Acta*, 1960, **23** (2), 186–188 (in English). —A rapid method for the determination of Cu in the presence of metals of groups 2 and 3 is described. *Procedure*—To 4 to 8 ml of a neutral or slightly acid soln. containing $\not<$2·5 mg of Cu add 4N-HCl (1 to 2 ml) and 6% aq. thioacetamide soln. (1 ml). Heat for 20 min. at 90°. Remove the ppt. by centrifuging and wash it thoroughly with water. Dissolve it in a few drops of 60% HNO_3, add glycine soln. (10% aq. containing 4% of Na citrate) (25 ml) and dilute to 50 ml. Measure the extinction in a 4-cm cell at 735 mµ. It may be necessary to

clarify the coloured soln. by centrifuging. The accuracy of the method is limited by the sensitivity of the colour measurement; the Cu^{II} - glycine complex is very stable. G. S. ROBERTS

934. **Disodium barium ethylenediaminetetra-acetate in complexometry; application to the determination of copper(II) and zinc(II).** F. Sierra Jiménez and C. Sánchez-Pedreño (Univ. Murcia, Spain). *Inf. Quim. Anal.*, 1960, **14** (4), 91–99.—The advantage of the disodium barium salt of EDTA over the disodium salt is that H^+ are not liberated during complex formation with metals, so that a buffer soln. is not required in the titrations. For the determination of Zn, the metal soln. is neutralised and, after addition of Eriochrome black T indicator and aq. K_2SO_4 soln., is titrated with the complexing agent. If Cu is present, aq. $Na_2S_2O_3$ soln. is added before the addition of the indicator; if Fe is present, solid Na tartrate is added to the original soln. Zinc, Cu or total Zn plus Cu can be determined with murexide as indicator. L. A. O'NEILL

935. **Photometric determination of small amounts of copper with picolinaldoxime.** H. Hartkamp (Inst. f. Spektrochem. und angew. Spektroskop., Dortmund-Aplerbeck, Germany). *Z. anal. Chem.*, 1960, **176** (3), 185–194 (in German).—The reagent forms a brownish-orange complex with Cu^+ at pH 4·5 to 6·5. *Procedure for weakly acid soln.*—To the sample (10 to 1000 μg of Cu) in a 100-ml flask are added 10 to 20 ml of buffer soln. [2N-acetic acid - 2N-Na acetate (1:4) (pH 5)], 2 ml of hydroxyammonium chloride soln. (10%) (**I**) and 5 ml of reagent (1% picolinaldoxime in methanol) (**II**). The soln. is diluted to 100 ml with water and shaken, and the extinction is measured at 422 mμ against a blank. The content of Cu is obtained from a calibration curve. In the presence of Cr, Fe, Co or Ni, 5 ml of tartaric acid (10%) is added and any ppt. (hydrolysis products of Sb, Bi, Ag or Hg) is filtered off before the reduction with **I**. *For alkaline soln.*—To the alkaline soln. (pH > 12) containing 10 to 1000 μg of Cu are added 5 ml of **II**, 2 ml of **I** and 5 ml of 5N-NaOH and the mixture is diluted to 100 ml with water. The colour intensity is measured at 445 mμ. The reaction is sensitive and the complex is stable for at least 24 hr. The effects of other elements are tabulated. B. B. BAUMINGER

936. **Spectrophotometric determination of copper following extraction with 1,5-diphenylcarbohydrazide [sym.-diphenylcarbazide] in benzene.** R. E. Stoner and W. Dasler (Med. Sch., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1207–1208.—Although not as sensitive as the analogous method of Turkington and Tracy (*Ibid.*, 1958, **30**, 1699), the procedure given ensures a colourless blank, almost instantaneous max. development of colour, and stability ($\simeq$2 weeks) of the reagent in benzene soln. (10%, w/v). *Procedure*—To an aliquot ($\equiv$ 0·5 to 3 μg of Cu) of the sample soln. in a centrifuge tube add satd. aq. Na_3PO_4 soln. (2 ml), dilute to $\simeq$5 ml with H_2O and add the reagent soln. (7 ml). Shake the tube vigorously for 1 min., and centrifuge for 2 min., then transfer the organic phase to a 1·5-cm cell and measure the extinction, against a similarly treated blank, at 540 mμ. Interferences (discussed) are not serious, but purines and pyrimidines must be absent. The standard deviation is 0·08 μg for 3 μg of Cu. In place of benzene, CCl_4, $CHCl_3$ or isoamyl alcohol can be used. W. J. BAKER

937. **X-ray fluorescence analysis and its application to copper alloys.** F. R. Bareham and J. G. M. Fox (Brit. Non-Ferrous Metals Res. Ass., London). *J. Inst. Met.*, 1960, **88** (8), 344–351.—A brief description of the principles and apparatus is given. Although the analysis is confined to superficial layers $\simeq$0·001 in. thick, a surface finish with 100 emery-paper did not affect the fluorescent radiation significantly if the emery lines were perpendicular to the axis of the spectrometer. Serious inter-element interference effects are explained and general procedures to avoid these are given. Copper concn. of 60 to 98%, with a coeff. of variation of $\simeq \pm$0·15%, have been determined in 1 min., exclusive of sample preparation. J. W. O. PYEMONT

938. **Photometric determination of gold in ores and concentrates.** J. Jankovský (Inst. Res. Ores, Prague). *Hutn. Listy*, 1960, **15** (9), 725–726.—*Procedures*: (*i*) *Ores with low SiO_2 content*—To the finely powdered sample (10 or 25 g) add conc. H_2SO_4 (50 or 100 ml) and heat on a sand bath, first gently, then to boiling. If necessary, add more H_2SO_4 (10 to 30 ml). After 3 to 4 hr. pour the liquid and undissolved residue into H_2O (300 or 600 ml), with stirring, and heat till the sulphates dissolve. Filter, wash the residue with hot H_2O, dry and ignite the filter in a porcelain crucible. Transfer the ash to a beaker, add aqua regia (10 ml) and NaCl soln. (20%) (3 drops) and heat for 10 min. on a water bath. Dilute with H_2O (10 ml), filter through a sintered glass filter, wash the ppt. with HCl (1:3) and evaporate the filtrate to dryness. (*ii*) *Ores with high SiO_2 content*—Treat 25 g of sample in a platinum dish with H_2SO_4 (1:1) (50 ml) and then HF (50 ml). Evaporate slowly on a sand bath with occasional stirring till SO_3 fumes appear. Cool, add conc. H_2SO_4 and boil for 1 to 2 hr. Pour the liquid into cold H_2O (500 ml) with stirring, boil for 10 min., add filter-paper pulp, boil and filter through paper. Proceed as in (*i*) except that, after dissolving the ash in aqua regia, evaporate the soln. to dryness, add conc. HCl (5 ml), repeat the evaporation, dissolve the residue in HCl (1:1) (10 ml) and filter. (*iii*) *Ores with high Sb content*—Proceed as in (*i*) or (*ii*) except that, after heating with conc. H_2SO_4, pour the liquid into HCl (1:4) containing a little $FeSO_4$. (*iv*) *Photometric determination*—Dissolve the evaporated residues by heating with HCl (1:1) (10 ml), transfer to a 100-ml separating-funnel, add KBr (5 g), dilute with H_2O to 50 ml and extract for 1 min. with ethyl ether (15 ml). Separate the aq. layer and repeat the extraction twice. Combine the extracts, add NaCl soln. (20%) (3 drops) and evaporate the ether. Dissolve the residue in aqua regia (3 to 4 ml) and evaporate on a water bath to dryness. Moisten with HCl (1:1) and repeat the evaporation. Dissolve the residue in N-HCl (5 ml), transfer to a 50-ml flask, add H_3PO_4 (1:3) (1 ml) and constant-boiling HBr (1 ml), dilute to vol. with H_2O and mix. After 10 min. measure the extinction in a Pulfrich photometer with filter S42. Carry out a blank and compare with a calibration curve. The results obtained in analysing samples containing 1·3 to 83·8 g of Au per tonne agreed with those obtained by the use of the fire-assay method. J. ZÝKA

939. **Gravimetric determination of beryllium with cinnamic acid.** I. Dema. *Rev. Chim., Bucharest*, 1960, **11** (8), 485–486.—The method proposed is to precipitate Be^{2+} at pH 5·6 to 6·5 with 10% ammonium cinnamate soln. in the presence of NH_4Cl.

Iron and Al must first be separated with ammonium carbonate; Li, Na, K, Ca, Sr, Ba, Mg, Mn, Ni and Tl do not interfere. H. SHER

940. **Determination of beryllium and estimation of impurities.** Report of U.K.A.E.A. Production Group Symposium, Blackpool (23.6.60). *Chem. Age*, 1960, **84**, 244.—This is a summary of the proceedings of the symposium.

941. **Spectrophotometric determination of beryllium and zirconium using chromotrope-2C [disodium o-carboxyphenylazochromotropate].** A. K. Majumdar and C. P. Savariar (Jadavpur Univ., Calcutta). *Z. anal. Chem.*, 1960, **176** (3), 170–174 (in English).—Micro amounts of Be and Zr give reddish-violet complexes with chromotrope-2C (**I**) at pH 6·0 and 1·5, respectively. The colour systems obey Beer's law from 0·025 to 0·375 p.p.m. of Be and 2 to 12 p.p.m. of Zr, and show max. absorption at 580 mμ. For practical purposes, 590 mμ is the best region, as at this wavelength **I** gives comparatively low absorption. The effects of pH, **I** concn. and time are discussed and the construction of calibration curves is described. Studies on the effect of **I** concn. have shown that, in a soln. of 25 ml, 2·5 ml of **I** soln. [0·05% (w/v) in water containing a few ml of methanol] is enough to produce max. colour intensity with 0·25 p.p.m. of Be or with 8·0 p.p.m. of Zr. The beryllium complex is stable for ≃15 min. and the zirconium complex for ≃4 hr. Interfering ions are listed. B. B. BAUMINGER

942. **Separation of beryllium from aluminium by ion exchange.** K. Vetejška and J. Mazáček (Inst. f. Erzforschung, Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2245–2247 (in German).—Beryllium and Al were separated by cation exchange, with resin FN, from soln. 0·5 to 0·7N in H_2SO_4. Beryllium passes through the column. The errors for mixtures containing Be and Al in ratios from 1:8 to 4:1 were $\ngtr$3·1%. B. HEINING

943. **Analytical use of dilituric acid. II. Determination of magnesium alone and in the presence of potassium.** M. Mára, R. Palous and V. Pavelka (Lab. Microbiol. and Immunol., Charles' Univ., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2240–2241 (in German).—Yellow dilituric acid is decolorised by the addition of magnesium salts. The method described is rapid. In the range of 0·025 to 2·0 mg of Mg per ml the relationship between change in Mg concentration and change in colour of the reagent is linear, and the mean error is <0·01 mg of Mg per ml. B. HEINING

944. **Analysis of slag from the manufacture of uranium metal. Determination of magnesium oxide and magnesium metal [in magnesium fluoride slag].** J. McKend (Eldorado Mining and Refining, Ltd., Tunneys Pasture, Ottawa, Canada). *Anal. Chem.*, 1960, **32** (9), 1193–1196.—In the procedure given, magnesium metal and MgO are leached from the slag (1 g) with a hot ammoniacal soln. of 0·1M-EDTA at pH 10, and the total Mg in the filtered extract after suitable treatment is determined by titration with 0·1M-EDTA, with Eriochrome black T as indicator. The magnesium metal is determined similarly in a separate sample, after extraction of the MgO by N-acetic acid (50 ml) in the presence of 10% $K_2Cr_2O_7$ soln. (15 ml). The MgO is obtained by difference. Provision is made for the removal of interfering ions (Fe^{3+}, U, Al^{3+} and F^-). The standard deviations are 0·03 and 0·13 for contents of 0·77% of Mg and 3·83% of MgO, respectively (8 determinations). W. J. BAKER

945. **Photometric determination of small amounts of calcium with glyoxal bis-(2-hydroxyanil).** F. Umland and K.-U. Meckenstock (Inst. f. anorg. Chem., Tech. Hochsch., Hannover, Germany). *Z. anal. Chem.*, 1960, **176** (2), 96–110 (in German).—Results are given on the variation in extinction of the complex of Ca with glyoxal bis-(2-hydroxyanil) (**I**) with NaOH concn. in 50% methanol, and it is shown that complex formation is complete in 0·04 to 0·12N-NaOH. The effects of anions and the behaviour of various cations are presented. *Procedure*—To a neutral soln. containing 150 to 300 μg of Ca is added 25 ml of N-HCl, and the vol. is made up to 200 ml with water. The soln. is heated to ≃80° and H_2S gas is passed through for 20 min., then the cooled soln. is diluted to 250 ml and centrifuged. To 25 ml of the supernatant liquid (15 to 30 μg of Ca) is added 5 ml of 0·1N-Na K tartrate, and the mixture is evaporated to ≃10 ml and neutralised with 0·1N-NaOH. If Be, Mg, Sr, Ba and U are present in larger amounts, 10 ml of 2N-NaOH, 5 ml of 0·1N-Na_2CO_3 and, when Ni, Co and Fe are present, 5 ml of 0·5N-KCN are added. Then 2 ml of ethanolic **I** soln. (0·6%) is added and the Ca is extracted first with $CHCl_3$ - hexanol (1:1) (2 × 25 ml), and then with 20 ml of 0·01N-HCl. To the separated $CaCl_2$ soln. is added 5 ml of 0·1N-Na K tartrate and ≃20 ml of water, and the soln. is adjusted with 0·1N-NaOH to pH 6 to 7. Then 25 ml of 2 × 10^{-3}M-**I** in methanol is added, the soln. is diluted to 50 ml with water, and after 10 min. the extinction is read at 520 mμ against a Ca-free soln. B. B. BAUMINGER

946. **Rapid determination of calcium as the oxalate in the presence of aluminium, iron and other impurities.** H. L. Watts (Aluminum Co., East St. Louis, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1189–1190.—To avoid a preliminary separation of the mixed oxides of Fe, Al and Ti, these and other elements are complexed with sodium gluconate (20% soln.) and then do not interfere in the ammoniacal pptn. of Ca oxalate at ≃100°. The ppt. is set aside for ≃30 min. before dissolution in H_2SO_4 (1:1) and subsequent titration with $KMnO_4$. If more than ≃10% of Mg is present in the sample, a double pptn. is necessary. Strontium and Mn are not usually present in sufficient concn. in limestone, dolomite or sinter-kiln feed to affect the results. On 18 samples containing from 25 to 50% of CaO the standard deviation was ≃0·08. W. J. BAKER

947. **Rapid titration of calcium in tricalcium phosphate with EDTA.** A. D. Ince and W. A. Forster [Albright & Wilson (Mfg) Ltd., Oldbury, Birmingham, England]. *Analyst*, 1960, **85**, 608–609.—Tricalcium phosphate (≃2 g) is boiled in a covered vessel with 25 ml of HCl (1:4) for 1 min. The cooled soln. is adjusted to 250 ml and dil. HCl (15 ml) is added to a 25-ml aliquot. After dilution to ≃250 ml, 50 ml of EDTA soln. (18·6 g of the disodium salt per litre) and 10 ml of aq. NH_3 are added and the excess of EDTA is titrated with standardised $ZnCl_2$ soln. (≃0·1N) to Eriochrome black T. For any one determination the difference between the result by the proposed method and that found gravimetrically may be expected to vary between +0·56 and −0·04% as CaO (95% confidence limits). A. O. JONES

948. **Separation of strontium from calcium in the presence of EDTA by the ion-exchange method.** O. Budevski and N. Krasnobaeva. *Compt. Rend. Acad. Bulg. Sci.*, 1960, **13** (1), 67–70.—*Procedure*—Use a column (30 cm × 9 mm) containing the cationite Wofatit F (NH_4^+ form) (10 cm, grain size 0·10 to 0·25 mm); wash with 0·02M-EDTA (ammonium salt) (**I**) at pH 5·1 ± 0·1 until the percolate is at the same pH. Add 0·02M-**I** (25 ml) to the test soln. (2 to 3 ml) containing 0·3 to 3·0 mg of Sr, adjust the soln. to pH 5·1 ± 0·1 and pass it through the column at the rate of 2 ml per min. Elute the Ca with 0·02M-**I** (150 to 180 ml) at pH 5·1 ± 0·1. Elute the Sr retained in the column with 0·1M-**I** (20 ml) at pH 10. A fraction collector is described consisting of 18 interchangeable test-tubes, each tube being calibrated at 10 ml and changed every 5 min. A recovery of 100% of Sr is obtained with a ratio of Sr to Ca of 100 to 1.

K. R. Cook

949. **Determination of strontium in rocks and biological materials.** B. A. Loveridge, R. K. Webster, J. W. Morgan, A. M. Thomas and A. A. Smales (A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1960, **23** (2), 154–171 (in English).—The determination of Sr by neutron activation and by stable isotope dilution is described and applied to a variety of rocks, minerals and botanical and zoological specimens. There is satisfactory agreement between results obtained by the two methods and an error of 1 to 4% of the Sr determined may be expected.

G. S. Roberts

950. **High-frequency titrations: estimation of barium, calcium and magnesium ions and determination of the hardness of water.** S. N. Mukherjee, P. K. Bhattacharjee and A. S. Battacharjee (Phys. Chem. Lab., Jadavpur Univ., Calcutta, India). *Sci. & Cult.*, 1959, **25** (2), 153.—Barium is titrated with standard sulphate soln. by the high-frequency technique previously described by Mukherjee (*Ibid.*, 1957, **23**, 53), the end-point being detected by a break in the capacity - concentration curve. The addition of an equal vol. of ethanol to the soln. is necessary for accuracy and precision. In the apparatus used (frequency 475 Mc/s) the valid range is from $8{\cdot}3 \times 10^{-3}$ to 2×10^{-3}M and the accuracy is within 0·8%. Barium and Ca may also be determined with standard oxalate soln. as titrant, the accuracy being within 1% and the ranges 1×10^{-3} to $3{\cdot}3 \times 10^{-3}$M and $1{\cdot}2 \times 10^{-3}$ to 5×10^{-3}M, respectively. In mixtures, Ba and Ca are titrated together with oxalate soln., and Ba alone with chromate soln. When present together, Mg interferes with the determination of Ca at a ratio of Ca to Mg of 1:10; Mg can be titrated with NaOH soln. over the range $8{\cdot}3 \times 10^{-4}$ to 5×10^{-3}M, the results agreeing with those by EDTA titration. In mixtures of Ca and Mg, both are pptd. by oxalate when the ratio of Mg to Ca is 1:10. If the ratio is less than this, only Ca is determined. In such cases, Mg is then determined in a second sample by titration with NaOH soln.; Ca does not interfere.

R. E. E.

951. **Determination of barium and strontium in crystals of sodium chloride and potassium chloride.** J. Trnka (Inst. Tech. Phys., Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2232–2234 (in German).—In the differential polarographic curve Ba, which gives a wave with $E_{\frac{1}{2}} = -1{\cdot}92$ V, can still be determined in concn. $\simeq 3 \times 10^{-5}$M. Strontium waves are less well developed, are considerably elongated and commence at a slightly negative potential. With a potential more negative than −2 V, reduction of the alkali metals occurs, and the recordings show frequent disturbances. Consequently the maximum galvanometer sensitivity cannot be used. The determination of both elements together is not possible.

B. Heining

952. **Evaluation of methods for trace zinc determination.** D. W. Margerum and F. Santacana (Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1157–1161.—Eight different methods for determining microgram amounts of Zn in the presence of an excess of other metals have been compared and evaluated. The efficiency of separation procedures was examined by radiochemical measurements of the distribution of ^{65}Zn between extraction phases. The preferred procedure is that described by Serfass and Levine (*Plating*, 1949, **36**, 818) and Butts *et al.* (*Brit. Abstr. C*, 1951, 287), in which masking of impurities by bis-(2-hydroxyethyl)dithiocarbamate (**I**) is followed by extraction of Zn with dithizone and measurement of the extinction of Zn dithizonate at 530 mμ. Pptn. of such hydroxides as $Fe(OH)_3$ is avoided by addition of CN^- or citrate; the excess of dithizone must be stripped with Na_2S soln. This method is rapid and, in the presence of a tenfold excess of Cd, Co, Cu, Hg, Ni, Pb, Fe, Mn, Cr or Sn, 30 μg of Zn can be determined with an accuracy of 99·0 ± 1·8% (duplicates, 95% confidence limits); 3 μg of Zn can be determined with satisfactory accuracy in the presence of a 100-fold excess of each metal. As a masking agent **I** is much more satisfactory than is diethyldithiocarbamate. The preliminary anion-exchange separation of Rush and Yoe's method (*Anal. Chem.*, 1954, **26**, 1345) is recommended for separating microgram amounts of Zn from milligram amounts of Co, Cu, Ni and Mn. (32 references.)

W. J. Baker

953. **5-Mercapto-1-phenyltetrazole as a reagent for zinc ions.** W. Dieterle and H. J. Becher (Lab. f. anorg. Chem., Tech. Hochsch., Stuttgart, Germany). *Z. anal. Chem.*, 1960, **176** (2), 118–121 (in German).—The method described is suitable for the detection and quant. determination of Zn^{2+}, which form a white ppt. with a 2% soln. of 5-mercapto-1-phenyltetrazole in methanol at pH 4 to 5. The limit of identification is 10 μg of Zn^{2+} per ml. Bivalent Mn does not interfere; Co^{2+}, Ni^{2+} and cations that form sulphides in HCl soln. should be separated.

B. B. Bauminger

954. **Extraction and amperometric titration of zinc and cadmium with sodium diethyldithiocarbamate.** Yu. I. Usatenko and F. M. Tulyupa (F. É. Dzerzhinskiĭ Dniepropetrovsk Chem.-Tech. Inst.). *Zavod. Lab.*, 1960, **26** (7), 783–786. *Determination of zinc and cadmium in aluminium alloys*—The sample (0·5 g) is dissolved in 15 ml of HCl (1:1), and the filtered soln. is treated with ≃1 g of K or Na citrate and then made slightly acid to methyl orange by means of aq. NH_3 and HCl. An aliquot (50 ml) of the diluted soln. (250 ml) is made alkaline by the addition of Na acetate and treated with 4 to 5 ml of 1% Na diethyldithiocarbamate soln. It is then shaken for 30 sec. with 15 ml of ethyl acetate, the aq. layer is treated with 1 to 2 ml of the diethyldithiocarbamate soln., and the extraction is repeated with 10 ml of ethyl acetate. The combined extracts are washed with water (3 or 4 × 10 ml) and the Zn and Cd are separated from other metals by re-extraction with 10 ml of 1·5N-HCl. The solvent layer is washed with water, and the

aq. extracts are treated with aq. NH_3 so that the final soln. contains ≮10% of NH_3. After addition of 1 ml of the Na diethyldithiocarbamate soln., Cd is extracted by means of ethyl acetate (2 × 10 ml). The Cd is back-extracted from the washed extract with 10 ml of 1·5N-HCl and titrated amperometrically in weakly alkaline soln., 0·1M in KCl, at +0·8 V with 0·01M-Na diethyldithiocarbamate. The ammoniacal soln. containing the Zn is neutralised with HCl and aq. NH_3, and an aliquot of the diluted soln. is titrated amperometrically with 0·01M-Na diethyldithiocarbamate. *Determination of zinc in copper-zinc sulphide ores*—The sample (0·5 g) is dissolved in 10 ml of conc. HNO_3, and aq. NH_3 is added until a ppt. forms, which is then dissolved in acid. An aliquot (10 ml) of the diluted soln. (250 ml) is treated with 0·5 to 1 g of Na acetate and 5 to 8 ml of 1% Na diethyldithiocarbamate soln., and then extracted with 15 ml of ethyl acetate. The procedure is then continued as described above. G. S. SMITH

955. **Polarographic determination of cadmium in ores.** L. T. Martȳnova and V. G. Sochevanov (All-Union Inst. of Mineral Raw Materials). *Zavod. Lab.*, 1960, **26** (7), 792–793.—The separation of Cd from large amounts of Cu, Ni, Fe and Al is achieved by adsorption of the Cl^- complex of Cd from 2 to 3N-HCl on the anionite ÉDÉ-10p, and subsequent elution with 0·05 to 0·1N-HCl. The Cd is determined polarographically in an ammoniacal basal soln. containing Cl^-. G. S. SMITH

956. **Amperometric determination of mercury with the rotating platinum electrode.** V. A. Zakharov, O. A. Songina and L. N. Terzeman (S. M. Kirov Kazakh State Univ.). *Zavod. Lab.*, 1960, **26** (7), 787–792.—Titration is carried out with KI soln. in a basal soln. of M-H_2SO_4, M-$(NH_4)_2SO_4$ or M-KNO_3, at +0·6 V *vs.* the N.C.E. In the presence of Fe^{3+} the initially high oxidation current decreases as the KI soln. is added, although I^- do not react with Fe^{3+}, which consequently do not interfere. In H_2SO_4 medium, Pb is pptd. as $PbSO_4$ and does not interfere, and the great difference between the solubility products of the iodides of Hg^{2+} and Tl enables Hg to be determined in the presence of Tl; Ag^+ and Cl^- must be removed. G. S. SMITH

957. **Isotopic analysis of boron in boron trifluoride by mass spectrometry and measurement of natural boron-10 concentration.** P. G. Bentley (U.K.A.E.A. Res. & Dev. Branch, Capenhurst, Ches.). *J. Sci. Instrum.*, 1960, **37** (9), 323–328.—"Memory" effects caused by adsorption of BF_3 on the surfaces of the inlet system of a mass spectrometer have been investigated with a wide range of concn. of ^{10}B. These effects are reduced by suitable design of the inlet system. Calibration of the instrument with BF_3 of known isotopic constitution showed that the true isotope ratios were lower by 3 to 4% than those measured; this is attributed to mass discriminations in the ion source. After correction for this error, the atomic concn. of ^{10}B in naturally occurring boron was found to be 19·27 ± 0·13%. G. SKIRROW

958. **Determination of boric oxide by the modified Wherry's method.** H. Bennett and W. G. Hawley (British Ceramic Res. Ass., Stoke-on-Trent, Staffs.). *Trans. Brit. Ceram. Soc.*, 1960, **59** (8), 315–322.—Appreciable errors have been discovered in the values for boric oxide in frits and glazes as determined by Wherry's process (Mellor and Thompson, "Treatise on Quantitative Inorganic Analysis," 2nd Ed., p. 656) and are due to variable alkalinity in the titration soln. after boiling off the CO_2; $CaCO_3$ is a strong factor in the development of alkalinity. These errors can be avoided by neutralising the alkalinity with HCl to methyl red before the addition of mannitol. D. C. ARMSWORTH

959. **Polarographic determination of aluminium, gallium and indium.** B. A. Cooney. *Dissert. Abstr.*, 1959, **19** (10), 2447.—The dye Superchrome Garnet Y (C.I. Mordant Red 5) reacts with Al, Ga and In forming chelates which are reducible at the dropping mercury electrode. This causes a negative shift in $E_{\frac{1}{2}}$, producing a polarographic wave, the diffusion current of which is a linear function of the concn. of metal in solution. Methods have been developed for the determination at pH 5·53 of Al, Ga, and Al-Ga and Ga-In mixtures and, over a limited range of concn., of Al-In mixtures. N. E.

960. **Colorimetric determination of titanium in once-refined aluminium [crude electrolytic aluminium]. Method with tiron (disodium 1,2-dihydroxybenzene-3,5-disulphonate).** B. Monlezun (Soc. d'Électro-Chimie d'Ugine, Lannemezan, France). *Chim. Anal.*, 1960, **42** (8), 377–380.—The metal is dissolved in aq. NaOH and oxidised with H_2O_2 and the soln. is filtered. Of the metals in the ppt., Cu, Mn and Fe interfere. The ppt. is dissolved in H_2SO_4 (1:10) (25 ml) and 5% aq. tiron soln. (2 ml) is added. Interference by Cu is eliminated by adjusting the pH to <7. The complexes of Fe and Mn are destroyed by the addition of glycollic acid (1 ml) and the Ti is determined colorimetrically at 440 mμ after 30 min. The yellow Ti complex is stable for several days. The method is 20 times as sensitive as the colorimetric method with H_2O_2. Concn. of Ti from 0·03 to 0·3% can be determined with an accuracy within 1%. G. BURGER

961. **Methods for the analysis of aluminium and aluminium alloys. Part 2. Determination of magnesium.** British Standards Institution (2 Park Street, London, W.1). B.S. 1728: Part 2: 1952. Amendment No. 2 (18.8.60).—The method for the determination of calcium is revised.

962. **Amperometric titration of gallium with cupferron solution.** I. A. Tserkovnitskaya, A. I. Kalinin and Yu. V. Morachevskiĭ (A. A. Zhdanov Leningrad State Univ.). *Zavod. Lab.*, 1960, **26** (7), 797.—Titration is carried out in 0·001 to 0·1N-H_2SO_4 with a vibrating electrode. Tartaric and citric acids do not interfere, but oxalic and lactic acids cause low results. Aluminium (≯50 times the content of Ga) and Zn (≯200 times) do not interfere. In the presence of Fe, reduction is carried out with Cd or $TiCl_3$ and Ga is extracted from the soln., 6N in HCl, with butyl acetate, and re-extracted with water. G. S. SMITH

963. **Complexometric titration (chelatometry). XLVIII. Complexometric determination of indium.** M. Kopanica and R. Přibil (Chem. Inst., Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2230–2232 (in German).—A slightly acid soln. of indium (2 to 50 mg) is diluted with water, and 10 to 20 ml of buffer (pH 3) (53·2 ml of conc. aq. NH_3, 90·4 ml of formic acid and water to 2 litres) is added. The soln. is heated to 50° to 60°, after the addition of 3 to 5 drops of indicator (9 g of 1,10-phenanthroline in the least possible vol.

of M-HNO_3, diluted to 1 litre), the soln. is titrated with 0·05M-EDTA (disodium salt) until the intense red - violet colour changes to yellow. Interference is caused by Fe^{3+}, Al^{3+}, Bi^{3+}, Pb^{2+}, Th^{4+}, Zr^{4+} and Ga^{3+}. B. HEINING

964. **Polarographic method of simultaneous determination of indium and cadmium.** Teiichi Matsumae (Government Ind. Res. Inst., Nagoya). *J. Electrochem. Soc. Japan (Overseas Ed.)*, 1959, **27** (10–12), E240–E241.—In N-KI the variance of the diffusion current of In increases with increase in concn. In soln. containing a constant amount of Cd, the diffusion current is proportional to the concn. of In and, in soln. containing a constant amount of In, it is proportional to the concn. of Cd. In the presence of SO_4^{2-} the diffusion current of In increases, and the $E_{\frac{1}{2}}$ shifts towards the negative side. This effect is greater in 0·1N supporting electrolyte. A similar effect, not so marked, is observed with Cl^-; NO_3^- have very little influence, and that of Fe^{3+} can be eliminated by reduction with hydroxylamine. Lead must be removed, as it dissolves in KI soln. to form PbI_4^{2-}, which give a reduction wave at approx. the same potential as that of In. In applications to zinc refinery products, good agreement is claimed with gravimetric results. R. E. E.

965. **Control of qualitative separation procedures with the aid of radionuclides. I. The behaviour of thallium.** E. Blasius and K. Riebartsch (Tech. Univ., Berlin-Charlottenburg 2, Germany). *Z. anal. Chem.*, 1960, **176** (5), 336–343.—In the investigation described, ^{204}Tl has been used to show that Tl, treated by usual qualitative procedures, appears in haphazard fashion throughout the first three analytical groups. In the amended procedure discussed, the separation is preceded by oxidation of all the Tl to Tl^{III} with $HClO_3$. In this case all the Tl, with the exception of those portions co-precipitated with H_2WO_4 and with Pt in the reduction-group, will reach the H_2S-group, where it may be quant. pptd. as the bromide. E. G. CUMMINS

966. **Separation of some rare earths by reversed-phase partition chromatography.** S. Siekierski and I. Fidelis (Department of Radiochemistry, Institute of Nuclear Research, Warsaw, Poland). *J. Chromatography*, 1960, **4** (1), 60–64 (in English).—A rapid method, carried out at room temp., is described for the separation of Ce, Nd and Pm; Ce, Pm, Sm, Eu, Gd and Tb; and Eu, Tb and Y. Tributyl phosphate is used as the stationary phase and the rare-earth metals are eluted with 11·5 to 15·8M-HNO_3 from columns of kieselguhr of diam. $\simeq$3 mm and bed length 11 cm. A flow rate of about 4 drops per min. is achieved by gentle pressure, and the radioactivity of every fifth drop is measured with an end-window Geiger - Müller counter. E. C. BUTTERWORTH

967. **Mis-identifications of the arc spectra of the rare earths.** R. N. Kniseley, V. A. Fassel and C. F. Lentz (Inst. for Atomic Res., Iowa State Univ., Ames, U.S.A.). *Spectrochim. Acta*, 1960, **16** (7), 863–880.—Errors in the lists of the emission lines of Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er and Yb, given in the MIT Wavelength Tables (1939), are due to impurities in the samples investigated. These errors (288) are listed in tables, together with the correct assignments as determined by photographing the spectra of highly purified rare earths in a spectrograph with a grating having 600 lines per mm. J. W. PRICE

968. **Titration of cerium(IV) sulphate with sodium oxalate at room temperature using ferroin as internal indicator.** V. Panduranga Rao, P. V. Krishna Rao and G. Gopala Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1960, **176** (5), 333–336 (in English).—The sample soln. (5 to 25 ml) is made normal with respect to H_2SO_4, diluted to 50 ml and 0·05N-$(NH_4)_2SO_4.Fe_2(SO_4)_3$ (2 to 5 ml) and 0·01M-ferroin (2 drops) are added. The mixture is titrated with 0·05N-Na oxalate, with magnetic stirring and exposure to the light from a high-pressure mercury-vapour lamp, the oxalate soln. being protected from the u.v. radiation. The end-point is indicated by the appearance of a vivid red colour with a brownish tinge. E. G. CUMMINS

969. **Use of azo dyestuffs for the polarographic determination of the lanthanides.** T. M. Florence and L. E. Smythe (Australian Atomic Energy Comm. Res. Estab., Lucas Heights, New South Wales). *Nature*, 1960, **187**, 771–772.—Some 2,2′-dihydroxy azo dyes, namely, Eriochrome violet B (C.I. Mordant Violet 5), Eriochrome black PV (C.I. Mordant Black 9) and C.I. Mordant Blue **31**, exhibit single polarographic reduction waves. In alkaline soln. they form chelates with the lanthanides, and the height of the wave due to the dye is reduced and a second wave appears at a more negative potential. The height of the second wave is proportional to the concn. of lanthanide. Eriochrome violet B was the most suitable dye for the determination of lanthanides. In ammonia buffer at pH 9, the difference in the half-wave potential for the free dye and the lanthanide complex increases with increasing atomic number, from 45 mV for lanthanum to 234 mV for lutetium. A. C. R. HARTLEY

970. **Separation of lanthanides by paper precipitation chromatography with 8-hydroxyquinoline. I. Separation and detection of lanthanum, cerium and praseodymium. I.** Hideo Hagai (Fac. of Sci., Univ., Kurokami-machi, Kumamoto). *Bull. Chem. Soc. Japan*, 1960, **33** (6), 715–717.—Single drops of binary mixtures of La - Ce, La - Pr and Ce- Pr as aq. soln. of the nitrates ($\simeq$10 mg per ml) were placed at the centre of circular filter-papers previously impregnated with 8-hydroxyquinoline and developed by irrigation from the centre. Solutions of acetic acid (**I**) and n-butyric acid (**II**) saturated with isobutyl alcohol were investigated as developers; Ce and Pr were partially separated with 0·01% soln. of **I**, and La - Pr and La - Ce with 0·5% soln. of **I**, but separations were impossible at higher acidities. Better separations were obtained with 1 to 3% soln. of **II**, the higher acidity minimising co-precipitation of the complexes. J. W. PRICE

971. **Colorimetric micro-determination of cyanide and thiocyanate.** G. S. Deshmukh and S. V. Tatwawadi (Benares Hindu Univ., Varanasi, India). *J. Sci. Ind. Res., India, B*, 1960, **19** (6), 195–198.—Cyanides and thiocyanates develop a yellow colour when treated with chloramine T in the presence of pyridine. From a study of the effects of concn. of reagents, pH and time of reaction the following procedure is recommended. To 1 to 3 ml of sample add 1 ml of 20% aq. pyridine and 1 ml of 0·1N-chloramine T. Make up to 10 ml, and measure the absorption after 2 to 10 min. in an absorptiometer with a violet filter with maximum transmission at 430 mμ. Read off the amount of cyanide from a calibration curve prepared from pure KCN or NH_4SCN. For amounts of KCN from 3·8

to 16·5 p.p.m. the errors ranged from −0·13 to +0·28 p.p.m.; for 1·7 to 22·1 p.p.m. of thiocyanate they ranged from −0·17 to +0·34 p.p.m. Bisulphate, nitrite and borate ions at concn. >40, 200 and 80 p.p.m., respectively, intensify the colour. Thiosulphate, iodide, sulphite, bisulphite and arsenite ions at concn. <1, 1, 20, 100 and 5 p.p.m., respectively, inhibit the reaction.

J. I. M. Jones

972. **Simultaneous determination of silicon and phosphorus in the presence of each other.** J. Paul (Dept. of Med. Biochem. and Pharmacol., Birmingham Univ., England). *Anal. Chim. Acta*, 1960, **23** (2), 178–182 (in English) —Phosphate and silicate are converted into phospho- and silico-heteropoly blue in aq. soln. The phospho-heteropoly blue is then extracted into ethyl ether and the extinctions of both the organic and the aq. phases are measured. *Procedure*—Transfer the test soln. (2 ml, containing 10 to 60 μg of SiO_3^{2-} and 50 to 400 μg of PO_4^{3-}) into a 50-ml separating-funnel and add N-HCl (2 ml) and 72% $HClO_4$ (1 ml). Shake for 1 min. then add 5% ammonium molybdate soln. (4 ml) and shake for 3 min. Add the reagent (0·2 g of 1-amino-2-naphthol-4-sulphonic acid, 2·4 g of $Na_2SO_3.7H_2O$ and 12 g of $NaHSO_3$, made up to 100 ml with H_2O) (0·5 ml) with continuous shaking. Set aside for 10 min. then extract with 20 ml of ethyl ether for 30 sec. Measure the extinction of the organic layer at 660 mμ and of the aq. layer (containing the silicon compound) at 690 mμ. The method is stated to be more sensitive for the determination of SiO_3^{2-} than for the determination of PO_4^{3-}; 12 μg of PO_4^{3-} per ml can be detected.

G. S. Roberts

973. **Determination of micro amounts of impurities in silicon by means of the γ-spectra of the radioactive isotopes.** K. I. Erokhina, I. Kh. Lemberg, I. E. Makasheva, I. A. Maslov and A. P. Obukhov (Phys.-Tech. Inst., Acad. Sci., USSR). *Zavod. Lab.*, 1960, **26** (7), 821–827.—The material is activated by neutron irradiation and the γ-radiation from isotopes of the impurities is analysed by means of a scintillation spectrometer. Very low concn. (several in the range 10^{-6} to 10^{-7} %) of Na, K, Mn, Cu, Zn, Ga, As, Mo, Ag, Cd, In, Sn, Sb, Ta, W and Au can be determined. G. S. Smith

974. **Routine spectrographic analysis for the determination of silicon in ferromanganese, and manganese, silicon and iron in silicomanganese.** A. Švehla (Kovohuty Istebné, Czechoslovakia). *Hutn. Listy*, 1960, **15** (8), 635–637.—For the determination of Si in ferromanganese, a polished massive sample is used. For the analysis of silicomanganese, the finely ground sample is mixed with powdered CuO and spectrally pure carbon (1:20:20). In both cases a copper upper electrode is used. Optimum results were obtained when determining 0·2 to 2·5% of Si in ferromanganese with the lines Si I 2881·58 and Mn I 2821·45 A, and 60 to 80% of Mn, 10 to 25% of Si and 4 to 20% of Fe in silicomanganese with the lines Si I 2532·38, Mn 2533·06 and Fe I 2535·60 A. J. Zýka

975. **Spectrochemical analysis of silicates using the Stallwood jet.** D. M. Shaw (McMaster Univ., Hamilton, Ontario). *Canad. Mineralogist*, 1960, **6** (4), 467–482.—A general spectrographic method for the simultaneous determination of 19 elements present in minor amounts in silicates, using a graphite matrix with Pd as internal standard, is described. The effects of irregular slit illumination, polar distribution of elements in the arc plasma, self-absorption, internal standards and the use of the Stallwood jet (*cf. Anal. Abstr.*, 1959, **6**, 3842) for d.c. spectrographic analysis are discussed.

R. A. Howie

976. **Determination of germanium by the molybdenum blue method.** Z. Řezáč and L. Růžičková (Res. Inst. for Inorg. Chem., Ústí n. L., Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2242–2244 (in German).—To a neutral soln. containing $\not>$250 μg of Ge add 15 ml of glacial acetic acid, and dilute with water to 40 ml. Add 1 ml of 10% ammonium molybdate soln., and, after 1 to 10 min., 1 ml of an aq. soln. containing $Na_2S_2O_5$ (15 g), $Na_2SO_3.7H_2O$ (1 g) and metol (0·5 g) in 100 ml. Shake the soln. after each addition, make up to 50 ml with water after 30 min. and measure the extinction at 827 mμ. The calibration curve is linear. The mean deviation is 2·1% (relative) (8 determinations). The time lapse between the molybdate addition and that of the reducing agent is critical. B. Heining

977. **Spectrographic determination of germanium in coal.** Ş. Mantea, M. Petrescu and V. Triţă. *Stud. Cercet. Metal., Bucharest*, 1959, **4** (4), 537–545. —The Ge is separated from the ash of the sample by fusion with K_2CO_3 - KNO_3, dissolution in aq. HNO_3, and distillation as $GeCl_4$. The distillate is treated with H_2S, $CuCl_2$ soln. is added, and the combined ppt. of GeS_2 and CuS is mixed with graphite, and with SnO_2 as internal standard (*cf.* Harris, *Anal. Abstr.*, 1954, **1**, 1803). The intensities of the lines Ge 2651·18 A and Sn 2661·25 A are compared. The dilution with CuS and SnO_2 is adjusted to give a spectrographic sample containing 0·005 to 0·1% of Ge. The reproducibility is within ±4% of the mean. Tests on known mixtures had a max. relative error $\not>$10%. A. R. Pearson

978. **Polarographic determination of germanium in solutions obtained by etching the surface of the material.** M. N. Platonova. *Zavod. Lab.*, 1960, **26** (7), 795–796.—The soln. used for etching contains H_2O_2 and either HCl or NaOH. After neutralisation to litmus paper the soln. containing Ge is made 2N in NaCl and the H_2O_2 is decomposed by the addition of 0·1 to 0·2 ml of a soln. 0·09N in $CuSO_4$ and 0·03N in $FeCl_3$, and 20 ml of a buffer soln. prepared by mixing 12 ml of 0·5M-KOH and 50 ml of 0·5M-H_3BO_3, followed by heating at between 80° and 90° for 10 min. The pH should be 8·2 to 8·3. After removal of O the polarogram is recorded over the range −1·2 to −1·6 V. G. S. Smith

979. **Polarographic determination of tin in stannic oxide.** S. L. Phillips (IBM Res. Lab., Poughkeepsie, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1062–1064.—The content of Sn in milligram amounts of stannic oxide can be accurately determined by dissolution of the sample in hot conc. (57%) HI in a Carius combustion tube, followed by polarography, at 25° under N, of the resulting soln. in 0·5M-$HClO_4$ - 2M-NH_4I as supporting electrolyte and 0·005% of gelatin as suppressor. For samples containing <20 mg of Sn, a supporting electrolyte of M-HCl - 4M-NH_4Cl with ascorbic acid (2 to 5 mg) is used. The wave of Sn^{4+} starts at approx. −0·4 V *vs.* the S.C.E. The error is within ±0·2 mg on 1 to 12-mg samples. W. J. Baker

980. **Polarographic determination of small quantities of lead and antimony in tin.** J. Kádár (Nat. Inst. of Science of Foodstuffs and Nutrition, Budapest). *Magyar Kém. Lapja*, 1960, **15** (7), 330–331.—In the method described, previous separation of the components is unnecessary. *Procedure*—The sample (0·1 g) is dissolved in 10% tartaric acid soln. (5 ml) and, after concentration (on a water bath), 20% HCl (5 ml) and solid $KClO_3$ (0·6 g) are added and the soln. is evaporated to dryness. The residue is taken up in conc. HCl (4 ml), solid Na_2SO_3 (1 g) is added and the mixture is heated under reflux on a water bath. When the reduction is complete the soln. is concentrated, then made up to 50 ml by the addition of 10% KCN soln. (5 ml) and 20% NaOH soln. Oxygen is removed by the passage of nitrogen for 8 to 10 min., and the polarogram is recorded in a basal soln. of N-aq. NH_3 - NH_4Cl containing 0·1% of gelatin. Evaluation is effected by the method of addition. The accuracy of the method is within ±5%, and the determination limit is 0·01%. Copper and arsenic do not interfere.
B. Vaszko

981. **Analytical chemistry of zirconium. I. o-Cresotic acid and 4-aminosalicylic acid in the determination of zirconium.** V. V. Subbanna and A. K. Bhattacharya (Univ., Saugar, India). *Anal. Chim. Acta*, 1960, **23** (2), 145–146 (in English).—Both *o*-cresotic acid and 4-aminosalicylic acid are found to be efficient precipitants of Zr, but *o*-cresotic acid gives a more readily collected ppt. and is less affected by pH change. *Procedure*—Buffer a soln. of Zr to pH 1·4 to 3·3 (4-aminosalicylic acid) or pH 1·4 to 5·4 (*o*-cresotic acid). Heat the soln. to 80° and add a 1% soln. of the reagent until pptn. is complete. Digest for 10 min., filter through Whatman No. 42 paper, wash the ppt. with hot water, dry and ignite to ZrO_2; Bi^{3+}, Cr^{3+}, Ce^{4+}, Mn^{2+}, Mo^{6+} and V^{4+} do not interfere. Double pptn. is needed if Fe^{3+}, Cu^{2+} or UO_2^{2+} are present. Thorium is pptd. quant. at all pH values.
G. S. Roberts

982. **Spectrographic estimation of hafnium and zirconium in their binary oxide mixtures.** K. S. Rajan and J. Gupta (Nat. Chem. Lab., Poona). *J. Sci. Ind. Res., India, B*, 1960, **19** (9), 341–344.—The intermittent a.c. arc is used for the determination of the Hf/Zr and Zr/Hf ratios. The lines chosen are Hf II 2641·4 Å, 2606·3 Å, 2665·9 Å and 2706·7 Å, and Zr II 2643·4 Å, 2678·7 Å, 2700·1 Å and 2722·6 Å in suitable combinations. Samples (5 mg) are arced in the oxide form between carbon electrodes. A Littrow-type large quartz spectrograph is used. The coeff. of variation of 6 determinations over the range 0·03 to 30% of Hf and over the range 0·1 to 90% of Zr was ≃5%. Iron and Ti interfere only when present in large amounts.
H. A. Foner

983. **Quercetin - hydrogen peroxide method for the colorimetric determination of hafnium in zirconium.** E. Cerrai and C. Testa (Lab. CISE, Milan). *Energia Nucleare*, 1960, **7** (7), 477–487 (in English).—The extinction of a soln. of the Zr - quercetin complex, but not that of the corresponding Hf complex, is lowered by the addition of H_2O_2. The total molarity of a soln. of Zr and Hf is determined colorimetrically or complexometrically, and then a suitable aliquot is adjusted to the correct acidity and treated with quercetin reagent (0·1% in ethanol) and after 5 min. with H_2O_2. After 30 min. the extinction of the soln. is measured at 425 mμ against a reagent blank, and the concn. of Hf and Zr are evaluated from a calibration graph. Details are given of methods for determining Hf in zirconium in the ranges 0 to 100% (M), 0 to 20% (M) and 0 to 5% (M). Interference from a large number of cations is avoided by a separation of Hf and Zr with *p*-bromomandelic acid.
J. H. Waton

984. **Volumetric determination of thorium by ferrocyanide.** K. Zutshi and J. N. Gaur (Maharani's Coll., Jaipur/Rajasthan, India). *Z. anal. Chem.*, 1960, **176** (3), 161–162 (in English).—The study reported previously (*cf.* Gaur and Bhattacharya, *J. Indian Chem. Soc.*, 1954, **31**, 467) has been extended to the volumetric determination of Th by the ferrocyanide method. To $Th(NO_3)_4$ soln. (≡0·08 g of Th) is added an excess of 0·2M-$K_4Fe(CN)_6$ (**I**). The mixture is shaken thoroughly, left to stand overnight and filtered. The excess of **I** in the filtrate is titrated with 0·037M-$Ce(SO_4)_2$, with the 1,10-phenanthroline - Fe^{2+} complex as indicator. This method has a definite advantage over the gravimetric method.
B. B. Bauminger

985. **Complexometric titration of thorium using some azo dyes from chromotropic acid.** Sachindra Kumar Datta (Chem. Lab., Victoria Gov. Coll., Coochbehar, India). *J. Sci. Ind. Res., India, B*, 1960, **19** (5), 168–170 (in English).—1,8-Dihydroxy-2-(5- and 6-sulphonaphthylazo)naphthalene-3,6-disulphonic acids (SNADNS-5 and -6), and 2-(4-carboxy-3-hydroxyphenylazo)-1,8-dihydroxynaphthalene-3,6-disulphonic acid (CHPADNS) (**I**) can be used for the titration of Th with EDTA in the pH ranges 3, 2·5 to 2·8, and 2·4 to 3·0, respectively; Fe, Zr, Ce^{IV}, Sn, Ag, Cu, Al, phosphate, fluoride and oxalate interfere. **I** is not affected by Fe^{II}, and Th can be titrated to this indicator in the presence of Fe after reduction with ascorbic acid.
W. T. Carter

986. **Determination of thorium by monochromatic X-ray absorption.** J. H. Stewart, jun. (Y-12 Plant, Union Carbide Nuclear Co., Oak Ridge, Tenn., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1090–1092.—The technique described can be used to determine Th in liquid samples containing percentage quantities of impurity elements in concn. ranges of 100 to 15,000 μg of Th per ml. Solid samples may be analysed after dissolution. With this technique, matrix effects and direct interference from impurities are eliminated. Lead and U do not interfere, but Bi and Sr do, and an extraction of the Th with n-butyl phosphate to eliminate these interferences is described. Duplicate determinations can be completed within 25 min. and the limit of error for a single analysis is ±2·8% for 2000 μg of Th per ml of soln. (P = 0·95).
K. A. Proctor

987. **Conductimetric estimation of thorium in dilute thorium chloride.** P. G. Menon and D. Singh (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1960, **176** (4), 264–269 (in English).—Titrations of $ThCl_4$ (0·001, 0·0005 and 0·00033M) and $ThCl_4$ - $CeCl_3$ mixtures with oxalic acid, ammonium oxalate and $AgNO_3$ soln. are reported. For $ThCl_4$, only oxalic acid is satisfactory, the minimum error being +0·5%. For the mixtures, only the end-point corresponding to the complete pptn. of both metals is indicated.
E. G. Cummins

988. **Separation of thorium from the rare earths by means of the cation exchanger FN.** K. Vetejška (Inst. Metal. Res., Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (7), 1895–1900 (in

German).—The behaviour of Th^{IV}, La^{III}, Ce^{III}, Ce^{IV} and Nd^{III} on the cation-exchange resin FN has been studied as a function of H_2SO_4 concn. From soln. 1·5N in H_2SO_4, Th^{IV} is not retained, but the other elements are quant. adsorbed. In chloride soln. Th^{IV} and La^{III} have almost identical adsorption characteristics. The application of this procedure to monazite sands is described.

T. R. ANDREW

989. **Potentiometric determination of ammonia in copper ammonium electrolyte.** N. A. Marchenko and V. F. Sityuk (V. I. Lenin Kharkov Polytech. Inst.). *Zavod. Lab.*, 1960, **26** (7), 793–795.—The first potential jump in the potentiometric titration of 10 ml of electrolyte with 5N-HCl corresponds to the neutralisation of the free NH_3. Total NH_3 is determined by titration with methyl orange as indicator.

G. S. SMITH

990. **Spectrophotometric determination of azide with ferric perchlorate.** A. Anton, J. G. Dodd and A. E. Harvey, jun. (Arkansas Univ., Fayetteville, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1209–1210.—Present colorimetric methods of determining azide with ferric salts (Labruto *et al.*, *Ann. Chim. Appl.*, 1932, **22**, 319; Roberson and Austin, *Anal. Abstr.*, 1957, **4**, 3623) can be improved by using $Fe(ClO_4)_3$, which eliminates the distillation, does not precipitate Ba or Pb from their azides, and extends the range of concn. of azide that can be determined. *Procedure*—Add 25 ml of H_2O to 25 ml of 0·11M-$Fe(ClO_4)_3$ and adjust the pH to 2·2 with 0·1N-$HClO_4$ or NaOH. Add the sample soln. (containing $\not>$ 10 mg of azide), stir and dilute to 90 ml with H_2O. Check the pH, transfer the soln. to a 100-ml flask and dilute to the mark. Measure the extinction at 454 mμ against an $Fe(ClO_4)_3$ blank and calculate the azide concn. from the working curve (0·5 to 10 mg per 100 ml). The precision is $\pm$0·08 mg (10 analyses) and the relative error is $<$6%. Thiocyanate in concn. $>$1 mg per 100 ml interferes.

W. J. BAKER

991. **Source of serious error in the determination of nitrates by the phenoldisulphonic acid method and its remedy.** F. B. Hora and P. J. Webber (Dept. of Botany, The Univ., Reading, Berks., England). *Analyst*, 1960, **85**, 567–569.—Determination of NO_3^- in NH_4NO_3 by the phenoldisulphonic acid method gave results low by 50% or more. Replicate experiments showed that the addition, immediately before evaporation, of 2 drops (0·1 ml) of the 30% KOH soln. used subsequently for developing the colour resulted in complete recovery of NO_3^-. Experiments with plant water cultures containing NH_4NO_3 and with standard soln. of KNO_3 were satisfactory. Since the KOH added is converted into K_2CO_3 during evaporation, the phenoldisulphonic acid reagent should not be added until the evaporated residue has cooled to room temp. Excessive effervescence is thus avoided. It is suggested that the loss of nitrate-N occurs via nitroamine, which decomposes to N_2O.

A. O. JONES

992. **Rapid analytical methods for the examination of metals and inorganic raw materials. XI. Determination of phosphoric acid in naturally occurring iron phosphates by ion-exchange separation.** P. Povondra and D. Roubalová (Chem. Inst., Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (7), 1890–1894 (in German).—The ground sample (0·3 to 0·4 g) is dissolved in HCl (1:1) (10 ml), oxidised with HNO_3 (5 ml), and evaporated to dryness with HCl; silica is removed by filtration and the filtrate (150 to 200 ml) is adjusted to 0·6M with respect to HCl. The soln. is then passed through a column of Amberlite IR-120 and washed with 0·05M-HCl (100 ml). Phosphate is determined in the eluate and washings. Adsorbed metal ions are eluted with 4M-HCl and the eluate is made up to 250 ml; Fe, Mn, Mg and Ca are determined in aliquots of this soln.

T. R. ANDREW

993. **Activation analysis of trace metals in sodium tri[poly]phosphate. Use of Dowex A-1 for chemical separations.** S. Forberg and S. Lundgren (Roy. Inst. Technol., Stockholm, Sweden). *Anal. Chem.*, 1960, **32** (9), 1202–1203.—A rapid and convenient procedure for the activity determination of trace amounts of Cu and Ni in sodium tripolyphosphate is described; it is based on quant. selective adsorption of these two elements on a 10-cm column of Dowex A-1 at pH 5. The interstitial soln. (containing activities of ^{24}Na, ^{32}P and others) is displaced with inactive aq. HCl, and the strong activity of ^{56}Mn is removed from the ^{65}Ni by selective displacement with phthalate-buffered inactive Mn^{2+} soln. The γ-spectrogram of interest of the resin can then be recorded with only moderate radiation precautions.

W. J. BAKER

994. **Determination of hypophosphite and phosphite in the presence of phosphate.** T. D. Verbitskaya and N. K. Romanova. *Zavod. Lab.*, 1960, **26** (7), 818–820.—For determining PO_2^{3-} in the presence of PO_3^{3-} a modification of the method of Sastri and Kalidas (*Anal. Abstr.*, 1957, **4**, 1813) is described. The soln. is treated with twice the amount of $(NH_4)_2SO_4.Fe_2(SO_4)_3$ required for oxidation of PO_2^{3-} and 10 ml of HCl (1:1), and the diluted soln. (50 ml) is boiled for 20 min. The cooled soln. is treated with 15 ml of H_2SO_4 (1:1) and 3 to 5 ml of conc. H_3PO_4, and titrated with 0·1N-Na_3VO_4 in the presence of Na diphenylaminesulphonate. A photometric method based on the reaction of PO_3^{3-} with $(NH_4)_2MoO_4$ is also described. The soln., containing 50 μg of P per ml, is treated with 25 ml of water, 6·5 ml of 5N-H_2SO_4 and 5 ml of 5% $(NH_4)_2MoO_4$ soln. and boiled under reflux for $\not<$2 hr. The cooled soln. is diluted to 100 ml and the extinction is measured with a red filter. The determination of PO_3^{3-} by the iodimetric method of Jones and Swift (*Anal. Abstr.*, 1954, **1**, 63) is satisfactory.

G. S. SMITH

995. **Cathodic polarographic wave of tervalent arsenic in alkaline media.** W. B. Swann, J. F. Hazel and W. M. McNabb (State Univ., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1064–1065.—A well-defined polarographic wave, with $E_{\frac{1}{2}} = -1{\cdot}86$ to $-1{\cdot}89$ V at pH 11 to 12·6, is obtained by the reduction of AsO_3^{3-} at 30° in 0·1M-LiCl - 0·01M-LiOH electrolyte at the dropping mercury electrode. The diffusion current is proportional to As concn. in the range 1 to 6 $\times$ 10^{-4}M, so that the method can be used to determine the concn. of AsO_3^{3-} in a sample. No such polarographic wave is obtained when the electrolyte is 0·1M-NaCl - 0·01M-NaOH.

W. J. BAKER

996. **Photometric determination of antimony [in copper] with crystal violet.** K. Študlar and I. Janoušek (Závod. V. I. Lenina, Plzeň, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (7), 1965–1969 (in Russian).—*Procedure*—Dilute the soln. of the sample (containing $\not>$20 μg of Sb) in 8N-HCl with the same acid to 5 ml, add $FeCl_3$ soln. (0·5%) (3 drops), reduce, with stirring, with a 10%

soln. of $SnCl_2$ in 8N-HCl till the soln. becomes colourless, add $NaNO_2$ soln. (10%) (0·5 ml), set aside for 3 to 5 min., dilute with H_2O to 10 ml, add urea soln. (25%) (1 ml), dilute to 50 ml and add crystal violet soln. (0·1%) (1 ml). Extract with 20 ml and 10 ml of trichloroethylene and measure the extinction at 610 mμ. Compare with a calibration curve. The following elements do not interfere —Al, As^{V}, Bi, Ca, Cd, Ce, Co, Cr^{III}, Fe^{II}, K, La, Mg, Mn, Na, Ni, Pb, Pd^{IV}, Se^{IV}, Sn^{IV}, Te^{IV}, Ti, U, W, Zn and Zr. Gold, Hg and Tl must be absent as they react similarly to Sb. Results with an error up to ±7·5% were obtained in determining 0·0004 to 0·008% of Sb in copper after a previous separation by co-precipitation with hydrated MnO_2. J. ZÝKA

997. **Determination of iron, lead and arsenic in antimony sulphide.** G. Norwitz, J. Cohen and M. E. Everett (Frankford Arsenal, Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1132–1135. —Three improved procedures, each having high precision and accuracy, are described. Iron is determined spectrophotometrically at 500 mμ after dissolution of the sample (0·5 g) in boiling HCl, addition of tartaric acid to prevent hydrolysis of Sb, of hydroxyammonium chloride to form Fe^{2+}, and then of 0·2% 1,10-phenanthroline soln. and 50% aq. Na acetate soln. Lead is extracted from an ammoniacal tartrate soln. by a 0·02% soln. of dithizone in $CHCl_3$, and is then pptd. as $PbSO_4$ after removal of the solvent and dissolution of the residue in HNO_3 - H_2SO_4. A sample of 1 to 3 g can be used and there are no interferences. The As in a 0·1 to 0·5-g sample is determined spectrophotometrically at 660 mμ by the molybdenum-blue method after distillation in ≃9 to 11 min. at $\not>$95° (to prevent volatilisation of Sb) from a HCl - HBr soln. A suitable all-glass distillation apparatus is illustrated. W. J. BAKER

998. **Colorimetric determination of vanadium with benzoylphenylhydroxylamine.** D. E. Ryan (Dalhousie Univ., Halifax, N.S., Canada). *Analyst*, 1960, **85**, 569–574.—To the sample soln. (0·03 to 0·4 mg of V^{5+}) in 5 to 9M-HCl is added a 2-ml portion of *N*-benzoyl-*N*-phenylhydroxylamine reagent (**I**) (0·5% w/v in $CHCl_3$) for each 0·1 mg of V expected, and the organic layer is diluted to ≃10 ml with $CHCl_3$. After separation, the aq. layer is extracted with $CHCl_3$ (10 ml). The combined extracts are diluted to 50 ml with $CHCl_3$ and the extinction is measured at 530 mμ or with an Ilford No. 605 filter. The standard soln. is prepared by dissolving $NaVO_3$ in 1% H_2SO_4, diluting the soln. to a known vol. and determining the V content gravimetrically in an aliquot with cupferron, suitable dilutions being then used. Steel samples (≃1 mg of V) are dissolved in 75 ml of H_2SO_4 (1:4) and a few drops of HNO_3. To an aliquot of the soln. is added $KMnO_4$ soln. until its colour just persists, the soln. is made 5 to 9M in HCl and extracted with **I** as before. Chromite and chrome-magnesite refractory are fused with Na_2CO_3 and $Na_2B_4O_7$, the melt is dissolved in water, $Cr_2O_7^{2-}$ are reduced by addition of $FeSO_4$ soln., $KMnO_4$ soln. is added until its colour persists, and the extraction and determination are completed as before. Vanadium must be present as V^{5+}. A. O. JONES

999. **Amperometric determination of quadrivalent and tervalent vanadium when present together.** I. A. Tserkovnitsa and N. A. Kustova. *Vestn. Leningr. Univ.*, 1960, No. 16, *Ser. Fiz. i Khim.*, (3), 148–149.—The determination is possible at a pH of 3 to 4, as vanadium salts form a stable complex with EDTA within that range; with a vibrating platinum electrode at +0·9 V *vs.* the S.C.E., and 0·1N-EDTA (disodium salt), a titration curve is obtained in which the first inflection corresponds to the V^{3+} content and the second to the V^{4+} content. K. R. COOK

1000. **Photometric determination of vanadium in hard metal, and carbides and oxides of tungsten, niobium, tantalum and titanium.** H. Schwarz (Gebr. Bohler and Co. A.-G., Kapfenberg, Steiermark, Austria). *Z. anal. Chem.*, 1960, **176** (4), 241–249.—For the determination of V in hard metal the finely powdered material (1 g) is converted into oxide by heating for 2 hr. at 800°, then fused with Na_2CO_3 for 30 min. at 900° to 1000°. The melt is dissolved in 100 ml of water, treated with $KMnO_4$ in the presence of a little ethanol, diluted to 500 ml and filtered. The first 30 to 50 ml of filtrate is rejected, and 125 ml of the remainder is transferred to a 200-ml flask to which 6 ml of 3% H_2O_2 soln. and 35 ml of H_2SO_4 (sp.gr. 1·28) are added. Air is bubbled through to remove CO_2, then 10 ml of 20% KF soln. is added, and the soln. is made up to volume with water. The extinction at 470 mμ is measured within 20 min. The value for V was found to be high with samples containing >1% of Ti in the presence of 10% of Ta, 10% of Nb and 75% of W. Correct readings were obtained, provided that the measurements were made within 20 min., by the addition of 10 ml of 20% KF soln. to the acid mixture. Two calibration curves must be prepared, one from pure sodium vanadate soln. and one from a soln. containing both sodium vanadate and tungstate. E. G. CUMMINS

1001. **Chemical analysis of vanadium concentrate.** S. Král (SONP, Kladno, Czechoslovakia). *Hutn. Listy*, 1960, **15** (9), 727–728.—*Procedure*—Dissolve the sample (5 g) in H_2SO_4 (1:1) (100 ml), oxidise by boiling with HNO_3 (1:1) (20 ml), dilute with H_2O (100 ml) and heat to dissolve sulphates. Filter off SiO_2, wash, ignite the filter in a platinum dish, weigh, evaporate with HF and determine SiO_2 by difference. Dilute the filtrate to 1 litre and titrate the V_2O_5 in an aliquot potentiometrically with $FeSO_4$ soln. after oxidation with $(NH_4)_2S_2O_8$. In another aliquot (400 ml) oxidise V in the same way, remove the persulphate by boiling, precipitate Al, Fe and Ti with aq. NH_3, dissolve in HNO_3, repeat the pptn. and separate Al from Fe and Ti by boiling with NaOH soln. and filtering. In the filtrate Al is determined gravimetrically as Al_2O_3. Dissolve the ppt. of $Fe(OH)_3$ and $Ti(OH)_4$, dilute to a known vol., divide into two portions and determine Fe with NH_4SCN, and Ti with H_2O_2 (after masking Fe with H_3PO_4) photometrically. Fuse a fresh sample (4 g) in a silver crucible with Na_2O_2, boil with H_2O, mix and filter off the oxides of Mn, Cu, Ni, Ca and Mg from the soln. containing chromate and phosphate. To an aliquot of the filtrate add H_2SO_4 and H_3PO_4 and determine Cr titrimetrically or photometrically. Precipitate phosphate as $AlPO_4$ from another aliquot, wash the ppt., then dissolve it in HNO_3 (1:1) (40 ml), add NH_4NO_3 and molybdate soln. and determine PO_4^{3-} gravimetrically or titrimetrically. Dissolve the pptd. oxides in hot HNO_3 (1:1), dilute to a known vol. and determine Mn in an aliquot titrimetrically with sodium arsenite soln. or photometrically after the oxidation with $(NH_4)_2S_2O_8$. In another aliquot separate Ag as AgCl and determine Ni and Cu polarographically

in a medium containing satd. Na_2SO_3 soln. (10 ml), satd. NH_4Cl soln. (10 ml), conc. aq. NH_3 (30 ml) and gelatin soln. (0·5%) (10 ml) in 100 ml. Boil the remainder of the soln., separate $Fe(OH)_3$ and $Ti(OH)_4$ with aq. NH_3 and determine Ca in the filtrate by titration with $KMnO_4$ soln. after pptn. as oxalate; determine Mg in the filtrate gravimetrically as $Mg_2P_2O_7$. For the determination of As_2O_3, dissolve the sample in aqua regia (40 ml), add H_2SO_4 (1:1) (30 ml) and heat. Cool, transfer with H_2O to a distillation flask, add KBr (1 g), hydrazine sulphate (5 g) and conc. HCl (100 ml), distil off $AsCl_3$ into a soln. of HCl and determine As^{3+} iodimetrically or potentiometrically with $KBrO_3$. Large amounts of As can be pptd. as As_2S_3 and then determined iodimetrically. The sulphide S is determined iodimetrically after burning the sample (1 g) in a stream of O at 1300° to convert the S into SO_2. Total S is determined gravimetrically as $BaSO_4$ after fusing the sample with Na_2O_2 in a nickel dish. Carbon is determined volumetrically in the Ströhlein apparatus after burning the sample at 1200° in a stream of O. J. Zýka

1002. **Methods of separation and determination of niobium and tantalum. A critical survey.** P. Münchow (Bundesanstalt f. Materialprüfung, Berlin-Dahlem). *ChemikerZtg*, 1960, **84** (15), 490–494; (16), 527–532.—A review with 153 references is presented. The methods of separation surveyed include precipitation, chromatography, extraction and chlorination, and those for the determination of Nb and Ta in the presence of each other include oxidation, reduction, photometric, polarographic and radiochemical methods and spectroscopy. G. Burger

1003. **Spectrographic determination of niobium and tantalum in tantalite-niobite.** R. Boudergues (Direction des Mines, Dakar, W. Africa). *Chim. Anal.*, 1960, **42** (9), 421–425.—When the concn. of Nb_2O_5 is 5 to 65%, Nb is determined by intermittent arc spectroscopy, with a max. error of $\simeq$6%. The internal standard used is Co_2O_3, and the lines chosen are Nb 2590·94 A and Co 2582·24 A. Iron influences the intensity ratio of Co to Nb in concn. up to 10% of Fe_2O_3, above which the effect is constant; the maximum permissible amount of Fe_2O_3 is 70%. Tantalum does not interfere at concn. of Ta_2O_5 <60% nor does Mn at concn. of oxide <20%. With the same Co line as standard, Ta in the range 20 to 60% of Ta_2O_5 is determined by means of the line 2603·57 A with an error of $\simeq$10%. J. H. Waton

1004. **Analytical determination of small quantities of oxygen in solids by activation.** H.-J. Born and N. Riehl (Inst. für Radiochem., Tech. Hochsch., München, Germany). *Angew. Chem.*, 1960, **72** (16), 559–562.—Concn. down to 10^{-8} g of O per g may be determined by the irradiation method described. Neutron irradiation of ^{6}Li provides the tritons, which are used to convert the ^{16}O to the short-lived isotope ^{18}F. Extension of the method to the analysis of liquids and gases is discussed. E. G. Cummins

1005. **Determination of oxygen content of uranium oxides with potassium dichromate.** L. Neumann (Dept. Nucl. Fuels and Radiochem., Inst. Chem. Technol., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2235–2236 (in Russian).—*Procedure*—To the dried sample ($\simeq$0·5 g) add a known amount of solid $K_2Cr_2O_7$ ($\simeq$0·5 g) and dissolve the mixture in H_2SO_4 (1:1) (10 ml) with gentle heating for not longer than 2 hr. Cool and determine the unchanged $K_2Cr_2O_7$ iodimetrically. J. Zýka

1006. **Determination of sulphur in chromium metal. A. By combustion in oxygen.** H. Rothmann, J. Engels and H. Wirtz (Ges. f. Elektrometall. m.b.H., Nürnberg, Germany). *Z. Erzbergb. Metallhüttenw.*, 1960, **13** (6), 263–268.—The use of a resistance furnace gives incomplete recoveries, and chemical methods are slow, but a rapid combustion method is possible by using a h.f. furnace. *Procedure*—Mix 0·2 g of finely powdered chromium with 1 g of reduced iron and 0·4 to 0·5 g of tin turnings in a crucible (wt. $\simeq$5 g), and place in a combustion tube in the furnace. Pass a current of O (0·8 to 1 litre per min.) leading the exit gas through a polyethylene tube containing a valve into a titration vessel containing H_2O_2. Switch on the furnace and increase the O flow as the sample burns. Reduce the gas flow and continue it for a few minutes after combustion is complete. Titrate the H_2SO_4 formed with NaOH soln. Standardise the NaOH soln. against a steel of known S content or against 0·02N-H_2SO_4. A small blank due to the reduced iron was evaluated by carrying 10 successive blanks through the procedure. The high temp. of the combustion ($\simeq$1800°) can cause liberation of S from the crucible. The time required is 10 min. and the reproducibility is ±0·001% of S. The method can be used for the determination of S in ferrochrome and in chromic oxide.

B. By Horak's method.—The evolution method of Horak (*Anal. Abstr.*, 1954, **1**, 687) was tested and the results obtained by different laboratories were examined statistically. Good reproducibility (±0·0015%) was found after modification of the method as follows. (*i*) Instead of iodimetric titration of CdS soln., convert it into copper sulphide, filter, oxidise with H_2SO_4 and HNO_3 and determine the Cu photometrically after addition of aq. NH_3. (*ii*) Remove traces of S compounds from the phosphoric acid before use, either by oxidation with H_2O_2 and pptn. as $BaSO_4$ or, preferably, by distilling 200 ml with 5 g of tin turnings as in the original method. (*iii*) Use an air bath instead of an oil bath for the distillation to ensure that a sufficiently high temperature is reached for the complete reduction of sulphate to sulphide. J. W. Price

1007. **Collection of sulphuric acid mist in the presence of a higher sulphur dioxide background. Comparison of the electrostatic precipitator, the Greenburg - Smith impinger, and the sintered-glass filter.** J. V. Kerrigan, K. Snajberk and E. S. Anderson (San. Engng Res. Lab., Univ. Calif., Richmond, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1168–1171.—A systematic study shows that these instruments yield reproducible results over the range of 0·5 to 150 mg of sulphuric acid mist per cubic metre, and that these instruments are as efficient at low as at high concn. K. A. Proctor

1008. **Non-destructive analysis of selenium by neutron activation followed by gamma-ray spectrometry.** Minoru Okada (Gov. Chem. Ind. Res. Inst., Shibuya-ku, Tokyo). *Nature*, 1960, **187**, 594–595.—Neutron irradiation of natural selenium produces, among other isotopes, ^{77m}Se, which has a half-life of 17·5 sec. and a comparatively large cross-section. A rapid detection of Se is possible by irradiation of a sample at a flux of 3×10^{11} neutrons per sq. cm for 20 sec., cooling, and counting. The spectrogram

obtained has one peak with γ-ray energy in the range 0·15 to 0·17 MeV and half-life 17·5 sec. The ^{77m}Se peak is distinguished from radionuclides with similar γ-spectra by the half-life. Quantitative determination is possible for concn. between 0·1 and 100 μg by comparison of the photopeak height with that of standards. A. C. R. HARTLEY

1009. **Spectrophotometric determination of tellurium(IV) with 1,1′-dianthrimide.** O. B. Skaar and F. J. Langmyhr (Univ. of Oslo, Chem. Inst. A, Blindern, Norway). *Anal. Chim. Acta*, 1960, **23** (2), 175–177 (in English).—A soln. of Te^{IV} and 1,1′-dianthrimide in conc. H_2SO_4 gives absorption max. at 550 and 600 mμ after heating at 70° for 16 hr. Germanium and B react similarly; Br^- and F^- interfere. G. S. ROBERTS

1010. **Spectrophotometric determination of tellurium(IV) by extraction with tri-n-butyl phosphate.** Seiichiro Hikime (Fac. Sci., Hokkaido Univ., Sapporo). *Bull. Chem. Soc. Japan*, 1960, **33** (6), 761–765.—The yellow complex formed by Te with thiourea in 0·1N-HCl is extracted into tri-n-butyl phosphate (**I**) in the presence of a large excess of KSCN. Conditions for the extraction and determination of Te are examined. It is found that the addition of 2 ml of 8% thiourea soln. and 2 ml of 50% KSCN soln. to the sample soln. (20 ml) is adequate for the extraction of 1 to 500 μg of Te with 20 ml of **I**. The complex is completely extracted at pH 1. The extinction of the **I** phase is measured at 400 mμ. The effect of various ions on the extinction of the complex is examined, and a pre-extraction procedure for the removal of interfering ions is discussed. I. JONES

1011. **Studies in polarographic analysis. II. Amperometric titration of tellurite with potassium permanganate.** I. M. Issa, R. M. Issa and M. G. Allam (Fac. of Sci., Cairo Univ., Egypt). *Anal. Chim. Acta*, 1960, **23** (2), 196–198 (in English).—The amperometric titration of tellurite in NaOH soln. (0·05 to 0·1N) with $KMnO_4$ soln. is found to be superior to potentiometric methods. Reversed L-shaped curves are obtained at −0·8 and −1 V and V-shaped curves at −1·4 V. The effect of telluric acid on the titration is discussed. G. S. ROBERTS

1012. **8-Hydroxyquinoline and 8-hydroxyquinaldine as reagents for the colorimetric determination of chromium.** J. P. Tandon and R. C. Mehrotra (Univ. Gorakhpur, India). *Z. anal. Chem.*, 1960, **176** (2), 87–90 (in English).—The method consists in extracting Cr^{III} hydroxyquinolinate (**I**) or Cr^{III} hydroxyquinaldinate (**II**) with $CHCl_3$ from the reaction mixture and measuring the colour at 425 mμ or at 410 mμ, respectively. To the sample (≃0·0208 g of Cr per litre) is added 4 to 5 ml of either reagent (prep. described) and the mixture is heated for ≃5 min. After the addition of 5 ml of water, heating of the soln. is continued for a further 5 min. The mixture is cooled, transferred to a 100-ml separating-funnel and **I** or **II** is extracted first with 10 to 15 ml of $CHCl_3$, and then twice with 5 ml of $CHCl_3$. To the separated $CHCl_3$ layers is added anhyd. Na_2SO_4 (4 to 5 g), and after 10 to 15 min. the soln. is diluted to 50 ml with $CHCl_3$ and the extinction measured. This method is applicable in the presence of Ce^{3+}, Ni^{2+}, W^{6+}, Mn^{2+}, Th^{4+}, Zn^{2+}, Te^{+} and Ba^{2+}, but Fe^{2+}, Fe^{3+}, U^{6+}, Cu^{2+}, Co^{2+} and Mo^{6+} interfere. B. B. BAUMINGER

1013. **Determination of microgram amounts of nitrate in chromic acid.** I. G. McDonald and A. Lench (Australian Defence Standards Lab., Maribyrnong, Victoria, Australia). *Analyst*, 1960, **85**, 564–567.—To render NO_3^- reducible to NH_3 by Devarda's alloy in the presence of H_2CrO_4, the Cr^{6+} are reduced with ethanol. The aq. soln. (100 ml containing ≯10 g of CrO_3) is warmed with HCl (30 ml) and ethanol (10 ml) for 10 min., 5 ml of ethanol is then added and, after 5 min., the excess of ethanol is removed by boiling. The liquid is then steam-distilled with 45 ml of 50% NaOH soln. until ≃150 ml of distillate is collected. The distillate is discarded, Devarda's alloy is added and a further 95 ml of distillate is collected. The NH_3 in the distillate is determined by Nessler's reagent. The reproducibility at a level of 10 μg of N is ±5% and at the 150-μg level it is ±1%. A. O. JONES

1014. **Determination of molybdenum in molybdates and their concentrates.** C. Gheorghiu and E. Radulescu-Grigore (Fac. Chem., C.I. Parhon Univ., Bucharest). *Rev. Chim., Bucharest*, 1960, **11** (7), 415–416.—The method is based on the formation of $[MoS_4][Cr(NH_3)_5Cl]$ after reduction of the mineral. The preparation of the reagent from liquid NH_3, metallic sodium, $(NH_4)_2SO_4.FeSO_4$ and anhyd. $CrCl_3$ is described. The red compound $[Cr(NH_3)_5Cl]Cl_2$ is obtained. *Procedure*—Approx. 1 g of the mineral is fused with fusion mixture. The soln. obtained is neutralised with HCl, treated with ammonium tartrate to mask the Al, Fe and W, or with EDTA to mask Zn, Co, Mn, Ni, Ca, Mg, Ba, Al and V, and then made slightly alkaline with aq. NH_3. After the passage of H_2S, an aliquot (20 to 80 ml) is treated with a 3% soln. of the reagent in slight excess, with stirring for several minutes. The bright cryst. ppt. is collected immediately in a crucible, washed with 95% ethanol and ethyl ether, dried under vacuum and weighed. H. SHER

1015. **Determination of tungsten in niobium-containing alloys.** I. P. Kharlamov, P. Ya. Yakovlev and M. I. Lўkova (I. P. Bardin Central Sci. Res. Inst. of Ferrous Metallurgy). *Zavod. Lab.*, 1960, **26** (7), 786–787.—The ppt. of WO_3, Nb_2O_5 and SiO_2 obtained in the usual manner from 0·1 to 0·2 g of the alloy is ignited at ≯700° and fused with 2 g of K_2CO_3 - Na_2CO_3 at 850° to 900°. The melt is extracted with 80 to 100 ml of cold water and an aliquot (10 to 20 ml) of the filtered and diluted soln. (250 ml) is treated with 15 ml of 4% NaOH soln., 2 ml of 50% NH_4SCN soln. and 45 ml of HCl (2:1), free from HNO_3, and cooled to 14° to 16°. Then 5 drops of $TiCl_3$ are added and the soln. is shaken vigorously. This operation is repeated three times at intervals of 2 to 3 min., after which the soln. is set aside for 8 to 10 min. The soln. is diluted to 100 ml with HCl (2:1) and the extinction is measured at 420 mμ. A blank is carried out at the same time. Results are referred to a calibration curve. Results are quoted for samples containing from 0·7 to 3% of W. G. S. SMITH

1016. **Spectrographic determination of trace amounts of arsenic in tungsten compounds.** T. J. Veleker (Sylvania Electric Products, Inc., Towanda, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1181–1182. —From 0·0002 to 0·006% of As in tungstic oxide can be determined to within ±5% by emission spectroscopy. The oxide is mixed with powdered graphite - ZnO buffer (containing 0·5% of Sb_2O_3 as

internal standard) and excited in a boiler-cap graphite electrode by d.c. arc, whereby As is volatilised and the W spectrum is depressed. The radiation from the cathode side of the discharge is spectrographed (30,000 lines per in. grating, 4100 to 5050 A, 20-μ slit), and the result is calculated from the working curve of log intensity ratios As 2288·12 A : Sb 2288·986 A *vs.* concn., a background correction being made on the As line. Samples of tungsten powder, tungstic acid, tungsten dioxide and ammonium paratungstate must first be oxidised to WO_3. W. J. BAKER

1017. **Rapid polarographic determination of tin in ferrotungsten, metallic tungsten and tungsten ores.** S. Král and V. Rett (SONP, Kladno, Czechoslovakia). *Hutn. Listy*, 1960, **15** (8), 638–639.—*Procedure for ferrotungsten and metallic tungsten*—Dissolve the finely powdered sample (2 g), in a platinum dish, in HNO_3 (1 : 1) (20 ml) and a suitable amount of HF. Then add H_2SO_4 (1 : 1) (10 ml) and heat to fuming. Cool, transfer with H_2O to a 100-ml flask, add conc. HCl (10 ml) and reduced or metallic iron (2 g), mix, and heat for 10 min. at 60° to reduce Fe^{3+} to Fe^{2+}, WO_3 to W_2O_5, and Sn^{4+} to Sn^{2+}. Add another portion of HCl (10 ml), metallic iron (2 g) and HCl (10 ml), and reduce again by adding hydrazine sulphate (1 g). Then cool, add gelatin soln. (0·5%) (10 ml), dilute to vol., filter off the W_2O_5 and register the polarogram of the filtrate. The procedure takes 100 min. *For tungsten ores and concentrates*—Fuse the finely powdered sample (2 g) with Na_2O_2 (5 g) in an iron crucible, transfer to a 200-ml flask with water, cool, dilute to vol., and filter. To a 100-ml portion of the filtrate add HCl and evaporate to 50 ml. Transfer to a 100-ml flask and proceed as described above. J. ZÝKA

1018. **Determination of elements in tungsten sulphide catalysts.** V. A. Klimova, E. G. Bereznitskaya and G. K. Mukhina (Inst. Org. Chem., Acad. Sci., Moscow, USSR). *Izv. Akad. Nauk SSSR*, 1960, **8**, 1520–1521.—Tungsten, S, C and H are determined gravimetrically in fresh and spent catalysts by combustion in O at 450° to 500°. Oxides of sulphur are absorbed on electrolytically pptd. Ag at 600°; CO_2 and H_2O on Ascarite and anhydrone, respectively. Tungsten remains in the sample boat as WO_3 and is weighed as such. A sample of 4 to 25 mg is used and the method takes 45 to 60 min. A precision of $\pm 0{\cdot}3\%$ is obtained. J. W. PRICE

1019. **New analytical methods in the technology of uranium.** K. F. Sporek (Pacific Yeast Products Inc., Wasco, Calif., U.S.A.). *Chem. Canada*, 1959, **11** (4), 66–72.—Small amounts of Cl^- in uranium compounds are determined by distillation of the sample with HNO_3 and titration of the distillate by Volhard's method; S^{2-}, SO_3^{2-}, SO_4^{2-}, AsO_4^{3-} and PO_4^{3-} do not interfere, but Ag and Hg cause low results; concn. $>0{\cdot}005\%$ can be determined. Soluble silicates are determined by extraction of the molybdosilicate complex from HNO_3 soln. containing $Fe(NO_3)_3$ and tartaric acid, and colorimetric measurement; U and V do not interfere; AsO_4^{3-} and PO_4^{3-} have a negligible effect at concn. $\simeq 40$ mg per litre, but at a concn. of 400 mg per litre PO_4^{3-} cause slightly low results, while AsO_4^{3-} cause results to be $\simeq 60\%$ low. The sensitivity is $\simeq 4 \times 10^{-4}$ g of SiO_2 per litre. Sulphate in uranium ores is pptd. as $PbSO_4$, which is then dissolved in ammoniacal EDTA soln. and titrated with soln. of lead or zinc salts, with Eriochrome black T as indicator; the method is applicable to samples containing $>0{\cdot}1\%$ of sulphate. Fluoride in uranium tetrafluoride is determined by dissolving the sample in NaOH - H_2O_2 soln. and passing the soln. through a cation-exchange column; Na and U are retained, while F^- pass through and are titrated as HF in the percolate. After removal of As by volatilisation as $AsBr_3$, PO_4^{3-} are pptd. with magnesia mixture, the ppt. is filtered off, and the Mg in the filtrate is titrated with EDTA (disodium salt) soln., with Eriochrome black T as indicator. Uranium is determined spectrophotometrically. R. E. E.

1020. **A new sensitive reagent for uranium. 2-(2-Thiazolylazo)-5-dimethylaminophenol (TAM). Application to the assay of rocks and solutions low in uranium.** E. Sørensen (Danish Atomic Energy Comm., Res. Establ., Risø, Denmark). *Acta Chem. Scand.*, 1960, **14** (5), 965–968 (in English).—Uranyl nitrate is extracted into isobutyl methyl ketone (**I**) from a soln. saturated with aluminium nitrate. Addition of the reagent gives an intensely coloured complex which is measured spectrophotometrically with a sensitivity of 0·1 μg in the range of 1 to 5 μg per ml. *Procedure*—For samples of rock, dissolve by fuming with HF and $HClO_4$ and, if necessary, fusing with $NaHSO_4$. Oxidise with 2 drops of H_2O_2 soln. (6%) and neutralise with aq. NH_3. Add, per ml of soln., 0·025 ml of aq. NH_3 (12M) and 2 ml of aluminium nitrate soln. [1000 g of $Al(NO_3)_3.9H_2O$ and 100 ml of H_2O maintained at 100° to prevent crystallisation]. Take 0·3 to 1 ml of **I** per μg of U expected and extract twice for 2 min., with exactly half of the **I** for each stage. Combine equal vol. of the two extracts and shake for 2 min. with a scrubbing soln. [5·4 g of KIO_3 and 350 g of $Al(NO_3)_3.9H_2O$ in 250 ml of H_2O]. Transfer 2·5 ml of the organic layer and exactly the same vol. of reagent soln. (dissolve 31 mg of reagent in 80 ml of pyridine, add 150 ml of water, cool, add 10·4 ml of HCl and dilute to 250 ml) to a stoppered tube and shake gently for 5 min. Centrifuge, and measure the extinction in a 1-cm cell at 575 mμ against a reagent blank. For uranium mother liquors, proceed as described above, but omit the extraction with the scrubbing soln. J. W. PRICE

1021. **Liquid - liquid extraction of uranium and plutonium from acetate solution with tri-iso-octylamine. Separation from thorium and fission products.** F. L. Moore (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1075–1079.—Micro amounts of U^{6+} (*e.g.*, ^{233}U tracer) or Pu^{6+}, or both, can be rapidly separated from Th, Na, K, Ba, Ca, Sr, Zr, Nb, Ru, Fe, Pa, Am and rare-earth elements by extraction for $\simeq 2$ min. from 0·5M-acetic acid with a 5% (w/v) soln. of tri-iso-octylamine (**I**) in xylene containing 3% (v/v) of 2-butoxyethanol as modifier. The U is stripped from the extract by shaking with M-ammonium bicarbonate; recoveries are $>99\%$. Macro amounts of U ($\simeq 5$ mg per ml) can be separated similarly by using M-acetic acid and a 20% soln. of **I** and stripping with M-ammonium bicarbonate. In both instances the vol. ratio of aq. phase to organic phase should be approx. unity. Uranium can thus be directly extracted from 0·5M-HNO_3 - 0·5M-acetic acid mixtures, provided that Ru, Zr and Nb are removed by washing the organic extract with 5M-HCl before stripping U from it with 0·1M-HCl. Recoveries of U are usually $\nless 98\%$. To obtain a sample soln. suitable for extraction and to increase the selectivity, a preliminary hydroxide

pptn. [with $Fe(OH)_3$ or Nb_2O_5 as carrier for micro amounts of U], followed by dissolution in acetic acid, is recommended. Applications in radiochemical analysis, *e.g.*, separation of U and Pu from fission products, and in uranium metallurgy are outlined. W. J. BAKER

1022. **Anion-exchange studies of uranium(VI) in sulphate and carbonate solutions. Separation from mixtures.** S. M. Khopkar and A. K. De (Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1960, **23** (2), 147–151 (in English).—A systematic study is described of the anion-exchange behaviour of U^{VI} in sulphate and carbonate systems on Dowex 21K, with soln. of HCl, HNO_3, H_2SO_4, $HClO_4$, NaCl, NH_4Cl, $NaNO_3$ and Na_2SO_4 as eluting agents. Uranium was separated from Ce, Zr, Cs, Ag, Cd and V in the sulphate system and from PO_4^{3-} and MoO_4^{2-} in the carbonate system. G. S. ROBERTS

1023. **Spectrophotometric determination of small amounts of uranium with 8-hydroxyquinoline.** Kenji Motojima, Hiroyuki Yoshida and Kimie Izawa (Atomic Energy Res. Inst., Naka-Gun, Ibaraki-Ken, Japan). *Anal. Chem.*, 1960, **32** (9), 1083–1085.—The method is based on the quant. extraction of U^{VI} 8-hydroxyquinolinate with $CHCl_3$ at pH 6·5 to 9 and formation of the stable coloured complex $UO_2(CO_3)_3^{4-}$. Beer's law is obeyed for 2 to 40 μg of U per ml of soln., and most metal ions, in concn. $\not>$100 to 200 μg of each, do not interfere. *Procedure*—Add 1% 8-hydroxyquinoline soln. (3 ml) and 2N-NH_4Cl (5 ml) to the slightly acid sample soln. ($\simeq$70 ml, $\equiv$40 to 800 μg of U). Adjust the pH to between 8·5 and 9 (aq. NH_3), dilute the soln. to $\simeq$100 ml and extract with $CHCl_3$ (20 ml). Divide the org. phase into two equal fractions, remove H_2O droplets from one fraction with anhyd. Na_2SO_4 (1 g) and shake the other fraction with 0·2M-ammonium carbonate (20 ml) for $\simeq$1 min. Measure the difference in extinction between the two extracts at 380 mμ in a 1-cm cell. When Th or rare-earth elements (5 to 10 mg of each) are present use 2% 8-hydroxyquinoline soln. (3 ml) and 5% EDTA (disodium salt) soln. (5 ml) to avoid interference. The accuracy is high. W. J. BAKER

1024. **Polarographic analysis. II. Polarography of uranium in sulphate solutions.** I. M. Issa, R. M. Issa and L. A. Shalaby (Nat. Res. Centre, Cairo, Egypt). *Z. anal. Chem.*, 1960, **176** (4), 250–256 (in English).—Experiments with U^{VI} in H_2SO_4 soln. (0·1 to 6M) and in the presence of Na_2SO_4, the results of which are contrary to those of Heal (*Trans. Faraday Soc.*, 1949, **45**, Part 1, 455) are described; U^{VI} in the concn. range 0·5 to 4mM was successfully determined in both M- and 4M-H_2SO_4.

III. Polarographic behaviour of uranium in phosphoric acid solutions. I. M. Issa, R. M. Issa and L. A. Shalaby. *Ibid.*, 1960, **176** (4), 257–264 (in English).—Experiments demonstrating the suitability of phosphoric acid as a supporting electrolyte for the determination of U^{VI} in the range 0·5 to 4mM are described. E. G. CUMMINS

1025. **Determination of uranium-235 by the isotope dilution method.** R. K. Webster, D. F. Dance, J. W. Morgan, E. R. Preece, L. J. Slee and A. A. Smales (A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1960, **23** (2), 101–115 (in English).—The precision of this method for soln. of uranium - aluminium alloys, containing Al $\simeq$2·5M, Hg $\simeq$0·02M and ^{235}U $\simeq$3·2 g per litre, has been determined. Soln. containing U with a ^{235}U content ranging from 50 to 93% were used. Natural uranium and ^{233}U were used as tracers, the latter being the most satisfactory. Uranium was separated from the mixture of sample and tracer by solvent extraction with tri-iso-octylamine and isotopic analyses were carried out by mass spectrometry. The analytical method is such that the final aliquots contain amounts of radioactive material which may be handled with relative safety under a well-ventilated hood, although dilution of the original alloy soln. would have to be carried out by remote handling. With ^{233}U as tracer, the mean precision (3 σ) of a single analysis for four soln. of different ^{235}U content was 0·83%. G. S. ROBERTS

1026. **Estimation of uranium in ores by a.c. polarography.** B. Breyer and J. R. Beevers (Dept. of Agric., Univ., Sydney, Australia). *J. Electroanal. Chem.*, 1960, **1** (5), 345–350 (in English).—After decomposition and dissolution of the sample (1 to 2 g, 0·03 to >1% of U_3O_8), the soln. is adjusted so as to have a final concn. of 0·5M in $HClO_4$. An aliquot is then submitted to a.c. polarography by the procedure described previously (Breyer *et al.*, *Aust. J. Chem.*, 1953, **6**, 188). The height of the first wave of UO_2^{2+} (at −0·18 V *vs.* the S.C.E.) is linearly proportional to the concn. of U over the range of 4×10^{-4} to 2×10^{-3}M. The second wave is unsuitable for analysis. The reproducibility is ±0·5%. A large excess of VO_3^- ($\not>$$7·5 \times 10^{-3}$M) can be tolerated, but Sb, Ti and Mo must be removed initially, as also must Cu and Fe if the ratios of Cu^{2+} to UO_2^{2+} and Fe^{3+} to UO_2^{2+} exceed 1:1 and 10:1, respectively. Oxygen must be excluded and filtration must be avoided during the preparation of the sample soln. W. J. BAKER

1027. **Determination of microgram amounts of fluorine by diffusion.** R. J. Hall (Inst. of Animal Physiol., Babraham, Cambridge, England). *Analyst*, 1960, **85**, 560–563.—The residue from the ashing of samples is dissolved in N-$HClO_4$, the pH is adjusted to $\simeq$6 with KOH soln. and the vol. is reduced to 2 to 2·5 ml by evaporation at 70°. The pH is adjusted to 3 with 0·2N-$HClO_4$ and the vol. to 2·5 ml. The HF is collected on a cylinder of filter-paper impregnated with KOH soln. and held on an adapter fixed inside the screw cap of a polyethylene bottle. The test soln. (1 ml) is placed in the bottle with 72% $HClO_4$ (2 ml) and the cap is inserted. Four bottles containing water in place of the sample are used as blanks. After diffusion at 60° for 24 hr. the paper is extracted with water (3 ml) in a 5-ml graduated tube heated in boiling water for 2 min. The cold soln. is made just acid to phenolphthalein with $HClO_4$ and diluted to 5 ml with water, and the pH is adjusted to 3. Standard vol. of NaF soln. (1 to 5 μg of F) are added to 2-ml amounts of the combined blank extracts at pH 3. To each soln. is added a 2-ml portion of buffered Na alizarinsulphonate soln. and, after 5 min., the yellow liquids are titrated with 0·001N-$Th(NO_3)_4$. The recovery ranged from 96·6 to 100%. A. O. JONES

1028. **Volumetric determination of fluorides with a zirconium salt solution and fluorescent indicator.** Z. Kolařík (Inst. f. anorg. Chem., Masaryk Univ., Brno, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2228–2229 (in German).—The method described can be used in the presence of low concn. (<0·2M) of SO_4^{2-}. To the sample (2 to 40 mg of F⁻) add 10 to 20 ml of buffer (3 vol. of N-NaOH and 2 vol. of 2N-chloroacetic acid) and 10

drops of 0·1% morin soln. in ethanol, dilute to 100 ml and adjust to give an ethanol concn. of 20%. Add solid NaCl in excess and titrate slowly with 0·05 to 0·1M-zirconium oxychloride soln. under u.v. light until a pale-green fluorescence appears. As the quantity of zirconium soln. is not exactly stoicheiometric, a factor has to be determined by titration of standard fluoride soln. The results obtained are reproducible with an error of $\simeq \pm 0{\cdot}9\%$.
B. HEINING

1029. **A polarographic method for fluoride.** C. P. Wallis (School of Dental Surgery, 31 Chambers Street, Edinburgh, Scotland). *Talanta*, 1960, **5** (1), 61–62.—Amounts of F^- from 2 to 10 μg can be determined by the increase in peak height of a polarogram of the *o*-nitrobenzenearsonic acid liberated from a complex with thorium by addition of the sample. The reagent contains 0·006% of *o*-nitrobenzenearsonic acid, 0·00025M-$Th(NO_3)_4$, 8 mg per litre of F as NaF, 0·3N-chloroacetic acid, 0·15N-NaOH and 0·05N-NH_4Cl. The addition of NaF to the reagent is necessary to eliminate a flat portion of the calibration curve; if it is omitted, the sample may contain 10 to 18 μg of F^-. One ml of the reagent is used with 8 ml of sample.
G. BURGER

1030. **Determination of small amounts of chloride by Volhard titration. Evaluation of operator determinate end-point error.** E. A. Burns and R. F. Muraca (Jet Propulsion Lab., California Inst. Technol., Pasadena, U.S.A.). *Anal. Chim. Acta*, 1960, **23** (2), 136–144 (in English).—An attempt is made to determine the magnitude of the error in analyses involving back-titrations and visual end-points caused by the varying visual sensitivity of the operators, and a mathematical treatment and an empirical method for its evaluation are presented. A procedure for its elimination is described, and is applied to the Volhard titration for the determination of up to 0·09% of Cl^- in ammonium nitrate, ammonium perchlorate and potassium perchlorate, with mean absolute errors of 0·0004%, 0·0007% and 0·0015%, respectively.
G. S. ROBERTS

1031. **Direct potentiometric determination of chlorides in concentrated [electrolytic] baths.** J. Pradilla-Sorzano (Univ. Ind. de Santander, Bucaramanga, Colombia). *Rev. Univ. Ind. Santander, Colombia*, 1960, **2** (1), 45–48.—Half-cells are made up of silver - silver chloride electrodes dipping into a standard soln. of Cl^- in 1 to 2M-H_2SO_4 and into a mixture of the test soln. and an equal vol. of standard $AgNO_3$ soln., and the e.m.f. of the completed cell is measured with a Beckman potentiometer (model G). A relationship is derived between the e.m.f. of the system, the solubility product of AgCl and the pCl of the test soln., which enables the Cl^- concn. to be calculated. G. H. FOXLEY

1032. **Analytical control of bromine during its manufacture.** J. M. Rafols Rovira (Servicios Quím. de Aprovechamientos Salineros S.A., Spain). *Inf. Quim. Anal.*, 1960, **14** (4), 100–106.—The Br^- content of residual liquors from the manufacture of NaCl is determined by oxidation to BrO_3^- with excess of NaOCl, elimination of the excess with Na formate, and iodimetric determination of the BrO_3^-. The Cl content of bromine is determined from the fall in density (resulting from the replacement of Br^- by Cl^-) of an aq. KBr soln. on treatment with the bromine. The H_2O content is determined by gravimetric measurement of the SO_4^{2-} formed on treatment of the sample with SO_2; HBr is determined by agitation with aq. KBr soln., removal of Br by a current of air and titration of the acid with 0·01N-NaOH. L. A. O'NEILL

1033. **Determination of fission-product iodine. Cation-exchange purification and heterogeneous isotopic exchange.** W. J. Maeck and J. E. Rein (Phillips Petroleum Co., Idaho Falls, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1079–1080.—In the method described, the sample is applied to a Dowex 50-X8 column to remove interfering activities and the percolate flows into a soln. of silver iodide. The ppt. formed after isotopic exchange of iodine is filtered off, dried and counted in a multichannel analyser. The over-all recovery is 97% with a coeff. of variation of 3·1% when standard ^{131}I is used. No determination of yield is required.
K. A. PROCTOR

1034. **Iron(II) derivatives of pyridyl-substituted sym.-triazines.** H. Diehl, E. B. Buchanan, jun., and G. F. Smith (Iowa State Univ., Ames, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1117–1119.—Reactions of Fe^{2+} with eleven pyridyl-substituted 1,3,5-triazines, and details of combining ratios and absorption spectra, are reported and discussed in respect of the possible use of any of these compounds as colorimetric reagents for Fe. Although tris-(4-ethyl-2-pyridyl)-1,3,5-triazine and tris-(4-phenyl-2-pyridyl)-1,3,5-triazine have higher sensitivities towards Fe^{2+} than has tris-(2-pyridyl)-1,3,5-triazine, the last-named compound is easier to prepare and is recommended for the colorimetric determination of Fe^{2+}. W. J. BAKER

1035. **Quantitative inorganic chromatography. V. Separation and determination of ferrous and ferric iron.** F. H. Pollard, J. F. W. McOmie, G. Nickless and P. Hanson (Dept. of Phys. and Inorg. Chem., Univ., Bristol, England). *J. Chromatography*, 1960, **4** (2), 108–116 (in English).—The sample soln. of mixed iron chlorides is chromatographed on a column of Amberlite IRA-400 resin saturated with 4M-HCl. Separation is obtained by eluting with 4M-HCl for Fe^{2+} and then with 0·5M-HCl for Fe^{3+}. The iron content of the eluate fractions can be determined spectrophotometrically with 2-nitroso-1-naphthol-4-sulphonic acid. Tabulated results show that it is possible to determine the minor constituent in mixtures of Fe^{2+} and Fe^{3+} in ratios between 1:100 and 40:1. Maximum accuracy is attained at ratios close to 1:1 and 5 to 20 mg of each valency state. When Fe^{II} is the major constituent, some conversion into Fe^{III} occurs.
S. M. MARSH

1036. **EDTA titration of total iron in mixtures of ferrous and ferric iron. Application to iron driers.** C. A. Lucchesi and C. F. Hirn (Sherwin-Williams Co., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1191–1193.—The method of Kinnunen and Winnerstrand (*Chemist Analyst*, 1955, **44**, 33) is modified so that the back-titration is made in an aq. ethanol soln. containing 50 to 60% (v/v) of ethanol. *Procedure for iron salts*—To the aq. soln. (50 ml $\equiv$ $\simeq$10 mg of Fe) add ethanol (150 ml) and adjust the pH to 2 or lower with HCl (1:3). Add 0·01M-EDTA (40 ml), neutralise with aq. NH_3 (1:3), adjust the pH to 9 with buffer soln. ($\simeq$10 ml), add Eriochrome black T (C.I. Mordant Black 11) indicator, and immediately back-titrate the uncomplexed EDTA with 0·01M-$ZnCl_2$ to the first appearance of a red colour. The titration must be

completed within 2 min. *Procedure for iron driers*—To the sample (0·2 g) add ethanol (100 ml) and HCl (1:3) (3 ml). Heat the mixture just to boiling and after cooling to $\simeq 25°$ proceed as described above. The error is $\simeq \pm 0·10$ mg for contents of 10 to 13 mg of Fe, and an analysis takes only $\simeq 30$ min. W. J. Baker

1037. **Spectrophotometric determination of iron and copper with methyl 2-pyridyl ketoxime and their simultaneous determination in mixtures.** D. K. Banerjea and K. K. Tripathi (Dept. of Inorg. and Anal. Chem., Ass. for Cultivation of Sci., Calcutta, India). *Anal. Chem.*, 1960, **32** (9), 1196–1200.—In the procedures given, the extinctions of the coloured complexes of Fe^{2+} and Cu^{+} with methyl 2-pyridyl ketoxime (0·5% aq. soln.) are measured at 525 and 410 mμ, respectively, in alkaline soln. (pH 10·5 to 11). The development of colour is instantaneous and its intensity is constant for more than 24 hr. Beer's law is obeyed (Fe 0·05 to 10 p.p.m., Cu 0·5 to 37 p.p.m.), with an optimum range of 1 to 4 p.p.m. for Fe and 6 to 12 p.p.m. for Cu. Most common anions do not interfere, and certain interfering cations can be masked by the addition of tartrate. The same colour is produced by Cu^{2+} because this is reduced to Cu^{+} by an excess of the reagent. Both elements can be determined simultaneously in mixtures having Fe to Cu ratios ranging from 1:13 to 6:1. *Procedure for mixtures of Fe and Cu*—To the sample soln. (5 ml) add 2N-H_2SO_4 (1 ml) and 5% aq. KI (5 ml) and set aside in the dark for $\simeq 10$ min. Add 0·05N-aq. $Na_2S_2O_3$ until the colour of the free iodine is discharged, adding two drops in excess; then add the reagent (5 ml) and dilute to $\simeq 20$ ml with H_2O. Adjust the pH to $\simeq 11$ with KOH soln. and dilute to 25 ml before measuring the extinctions in a 1-cm cell. The accuracy is high. W. J. Baker

1038. **Preparation of silver crucibles for determination of iron in silicates.** E. B. Buchanan, jun., and H. Diehl (Iowa State Univ., Ames, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1216.—A silver crucible (2 to 3 mm thick), with smooth inner and outer surfaces, can be prepared by periodic reverse-current electro-deposition of silver (90 to 150 g) for $\simeq 24$ hr. on a 50-ml nickel crucible in a bath of $AgNO_3$ (30·5 g), NaCN (36·5 g) and KNO_3 (112·5 g) per litre, followed by dissolution of the nickel with hot HCl (1:1). A fixed solid-silver rod is used as anode and, after deposition of a thin layer of silver, the cycle is adjusted to 2 amp. per 7 sec. (plating) and 4 amp. per sec. (stripping). The crucible can be used in place of platinum in determinations of Fe involving fusion of silicates. W. J. Baker

1039. **Rapid polarographic determination of iron and manganese in minerals.** M. Ionescu and D. Dumitrescu. *Rev. Chim., Bucharest*, 1960, **11** (8), 483–485.—*Procedure*—The mineral (0·2 g) is treated with 5 ml of HCl (1:1) and evaporated to dryness; the residue is treated with 5 ml of HNO_3 - HCl (3:1), and again evaporated to dryness. The residue is re-dissolved in 6 ml of conc. HCl. For minerals requiring fusion with alkali, the melt is dissolved in acid and oxidised with H_2O_2, and the excess of O is boiled off. In each case the soln. is diluted to 100 ml, and a 5-ml aliquot is treated with 5 ml of 0·5M-triethanolamine and 5 ml of 5M-NaOH. After vigorous shaking for 15 sec., 3 drops of Na_2SO_3 soln. are added with 4 drops of 0·5% gelatin soln., and the mixture is left for 0·5 to 2 hr. before polarographic determination at 0 to $-0·6$ V for Mn ($E_{\frac{1}{2}} = -0·49$ V) and then at $-0·4$ to $-1·2$ V for the Fe ($E_{\frac{1}{2}} = -1·08$ V). The curves obtained are compared with standards prepared under similar conditions. H. Sher

1040. **Determination of hydrogen in steel by the carrier gas method.** K. Abresch, W. Dobner and H. Lemm (August Thyssen-Hütte A.-G., Duisberg-Hamborn). *Arch. Eisenhüttenw.*, 1960, **31** (6), 351–354.—A discussion is presented of the advantages of the carrier gas method of determining H in steel, the diffusion of the gas through palladium, the apparatus and the de-gassing method being described. Two different temperatures were used; the diffusible H was removed below 1200°, and total H was determined in a sample melted in an induction furnace. The carrier gas was Ar; H was separated from other gases by means of a palladium spiral, and collected and measured volumetrically. Sources of error are indicated. W. H. Kemp

1041. **Determination of low carbon contents in transformer steel by freezing.** R. Freiwillig, F. Novák and J. Čadek (Res. Inst. Iron Metallurgy, Prague). *Hutn. Listy*, 1960, **15** (8), 632–635.—The sample is burnt in a stream of pure O with Pb_3O_4 at 1200°. The CO_2 formed is carried with the stream of O over a layer of glass wool impregnated with MnO_2 and then into a U-shaped capillary tube, immersed in liquid N or liquid air, where it freezes. The capillary tube is then warmed to normal temp. and from the CO_2 pressure the amount of C is calculated. An accuracy of ± 6 μg of C was attained. The method is suitable for samples containing $<0·05$% of C and takes $\not> 10$ min. A detailed description of the apparatus is given. J. Zýka

1042. **Isolation and analysis of carbides from austenitic steel alloyed with niobium and molybdenum.** O. Opravil, O. Káčerová, J. Pažitný and I. Svatik (Výzk. Ústav Zváračský, Bratislava, Czechoslovakia). *Hutn. Listy*, 1960, **15** (8), 628–631.—The best results for isolating the carbide phase from steel were obtained by the use of a 10% soln. of HCl in ethanol as electrolyte and electrolysing for 8 hr., at a current intensity $\not> 0·10$ amp. per sq. cm, at a temp. $\not> 5°$. Two methods were used for the determination of Nb—the pptn. of Nb_2O_5 with H_2SO_3 and the pptn. of Nb with tannin (suitable for samples containing W). J. Zýka

1043. **Rapid determination of nitrogen in alloy steel (Cr 18 Mn 8 Ni 5 N 2).** M. Šúcha (Vítkovické Železárny, VŽKG, Ostrava, Czechoslovakia). *Hutn. Listy*, 1960, **15** (9), 729–730.—*Procedure*—Dissolve the finely divided sample (1 g) in hot $HClO_4$ (1:1) (25 ml) and H_2SO_4 (1:1) (10 drops). Evaporate till red crystals begin to form. Dissolve the residue by boiling with H_2O (30 ml) and transfer the hot soln. to the distillation flask of the Parnas apparatus. Add KOH soln. (50%) (80 ml), and distil off the liberated NH_3 into H_2O (100 ml) containing Tashiro indicator and a small known excess of 0·01N-H_2SO_4. After the neutralisation of the H_2SO_4, add a further small portion and continue the distillation. The procedure is finished when an excess of 3 drops of 0·01N-H_2SO_4 produces no colour change. The distillation is accelerated by passing a stream of air or N through the apparatus. J. Zýka

1044. **The effect of the pH value of electrolyte solutions on the quantity and composition of isolated oxide inclusions in fully quenched Thomas steel.** E. Piper, H. Hagedorn and M. Fröhlich. *Arch. Eisenhüttenw.*, 1960, **31** (6), 355–358.—Quenched Thomas steel (6 to 7 g) containing Ca and Si is electrolysed at 10 mA per sq. cm for 19 hr. in 1 litre of a solution containing 5% of Na citrate, 1·2% of KBr and 0·6% of KI, the pH being adjusted to between 6·8 and 1·0 with citric acid or HCl. Isolation is carried out in a rectangular electrolytic cell (illustrated). The pH changes by $\ngtr$ 1 unit during the electrolysis. The oxide residues obtained at various pH values differ neither in quantity nor in composition. Alteration of pH from 6·8 to 2 is not accompanied by loss of oxide inclusions.
W. H. Kemp

1045. **New redox systems. II. Oxidation of cobalt(II) with iron(III) chloride in 1,10-phenanthroline solutions.** F. Vydra and R. Přibil (Chem. Inst., Czech. Acad. Sci., Prague, Czechoslovakia). *Talanta*, 1960, **5** (1), 44–52.—The conditions under which Co^{2+} can be quant. oxidised by $FeCl_3$ in the presence of 1,10-phenanthroline have been established by potentiometric and colorimetric studies. The potentiometric determination is favoured by a high concn. of 1,10-phenanthroline (at least 6 times the mol. concn. of the Co), a pH between 2 and 4 and a temp. of $\simeq 50°$. Determination of the intensity of the red colour (max. intensity at 510 mμ) also proved that the reaction was quantitative.
G. Burger

1046. **Spectrophotometric determination of cobalt by mineral acid.** Yuroku Yamamoto, Masayuki Tabushi, Toyokichi Kitagawa and Tsunenobu Shigematsu (Shigematsu Lab., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1960, **38** (4), 313–317 (in English).—The absorption spectra of Co^{2+} in HCl, $HClO_4$, H_2SO_4 and acetic acid soln. are reported from $\simeq 200$ mμ to 1300 mμ. Except in chloride soln., Co^{2+} may be determined by the absorption of the aquo-complexes at 510 or 1145 mμ. In chloride soln. the absorption max. of the aquo-complexes are 518 and 1160 mμ, and a chloro-complex is formed with max. absorption at 688 mμ. The dependence of the extinction at 688 mμ on chloride concn. and acidity is reported.
T. R. Andrew

1047. **The volumetric determination of nickel alone or in the presence of aluminium.** I. Grecu, V. Handelsmann and E. Curea (Fac. Pharm., Univ. Cluj, Romania). *Rev. Chim., Bucharest*, 1960, **11** (7), 413–414.—The proposed method is based on the formation by Ni^{2+} and SCN^-, in excess, of an insoluble complex with amidopyrine *viz*, $[Ni(C_{13}H_{17}ON_3)_2](SCN)_2$, and the titration of the excess of SCN^- with $AgNO_3$ soln. *Procedure*—The soln. of Ni^{2+} is acidified with 1 to 2 drops of conc. acetic acid, then treated with 25 ml of 4% amidopyrine soln. and an excess ($\simeq 50$ ml) of 0·1N-NH_4SCN. The mixture is diluted to 100 ml, mixed thoroughly for 1 to 2 min. and left for 15 min. for the green ppt. to settle. The soln. is filtered and titrated against a known vol. of 0·1N-$AgNO_3$, with $(NH_4)_2SO_4.Fe_2(SO_4)_3$ as indicator, to a colour change from blue to red. Errors vary from +0·39 to −0·64%. If Al^{3+} are present the Al is initially pptd. as $Al(OH)_3$ and the whole treated with an excess of amidopyrine ($\simeq 35$ to 40 ml). The limits of error are similar.
H. Sher

1048. **Nicotinamidoxime as an analytical reagent. III. Polarographic studies on the nature of the nickel complex and spectrophotometric determination of the metal.** K. K. Tripathi and D. Banerjea (Indian Ass. for the Cultivation of Sci., Jadavpur, Calcutta). *Z. anal. Chem.*, 1960, **176** (2), 91–96 (in English).—Nicotinamidoxime (**I**) gives a deep blue colour with Ni^{2+} in alkaline soln. on treatment with iodine, and the polarograms of the coloured soln. and of the separate components indicate that **I** is first oxidised to an azo-type of compound, which forms anionic complexes with Ni^{2+}. The extinction of the soln. is measured at 575 mμ for 0·3 to 10 p.p.m. of Ni against a water blank. The effects of iodine, pH, reagent concn. and foreign ions are shown. The nickel soln. in 8M-HCl is freed from interfering cations, then treated with **I** (0·25M); the pH is adjusted to $\simeq 11$, 5 ml of iodine soln. (0·05M) is added and the mixture is diluted to 25 ml with water. After 10 min. the colour of the soln. is measured. The sensitivity of the method is 0·014 μg of Ni per sq. cm, and the visual identification limit is 0·5 μg of Ni per ml. Most of the common anions are without effect, but cyanide and EDTA must be absent. Bivalent Ni is separated from Cu^{2+}, Co^{2+}, Zn^{2+}, Mn^{2+} and Fe^{3+} by the anion-exchange method of Kraus and Moore (*J. Amer. Chem. Soc.*, 1953, **75**, 1460). Interference from Pb^{2+}, Bi^{3+}, Al^{3+}, Ti^{4+}, Zr^{4+} and Th^{4+} is masked by the use of excess of tartrate.
B. B. Bauminger

1049. **Anodic stripping voltammetry of nickel at solid electrodes.** M. M. Nicholson (Humble Oil & Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1058–1062.—Results are presented for anodic stripping measurements on nickel in thiocyanate soln. at platinum and gold electrodes, with the application of a linear voltage scan during the stripping process. Reproducible results can be obtained for concn. of Ni as low as 3 in 10^9.
K. A. Proctor

1050. **Spectrophotometric determination of nickel by mineral acid.** Tsunenobu Shigematsu, Yuroku Yamamoto, Masayuki Tabushi and Toyokichi Kitagawa (Shigematsu Lab., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1960, **38** (4) 307–312 (in English).—The absorption spectra of soln. of Ni^{2+} in HCl, $HClO_4$, H_2SO_4 and acetic acid are reported from $\simeq 280$ mμ up to 1250 mμ. Except in the presence of Cl^-, Ni^{2+} may be determined by the absorption maxima of the aquo-complexes at 394, 720 or 1130 mμ. In chloride soln. the acidity and total Cl^- concn. must be kept constant as the absorption maxima vary with conditions.
T. R. Andrew

1051. **A critical review of methods of isolating and separating the six platinum metals.** F. E. Beamish (Dept. of Chem., Univ. of Toronto, Ontario, Canada). *Talanta*, 1960, **5** (1), 1–35.—All methods of assaying platinum-metal ores published up to Jan. 1960 are reviewed. A new fire-assay method, based on the reduction of the associated base metals by carbon, Na_2CO_3 and borax at 1450° to form a 25-g alloy button, is reported. (157 references.)
G. Burger

1052. **The application of anion-exchange techniques to the spectrochemical detection of the noble metals in silicate rocks.** R. R. Brooks and L. H. Ahrens (Dept. Physiol., Univ., Cape Town, S. Africa). *Spectrochim. Acta*, 1960, **16** (7), 783–788.—Gold and the platinum metals are concentrated to

the threshold of spectral sensitivity by ion exchange of their chloro-complexes on a resin column. A 150-g sample of dunite containing 3 p.p.m. of noble metals gave detectable lines of Au and all the platinum metals except Os; Au and Pt were detected in a 200-g sample of Cape granite, showing an enrichment factor of 20,000. *Procedure*—Treat the sample in a Teflon beaker with a mixture of HF and aqua regia and evaporate to dryness. Fume with H_2SO_4, oxidise with Br and dissolve in 2N-HCl. Adsorb on a column of Amberlite IR-400, 20 cm long and 1 sq. cm in cross-section, and elute with 2 litres of 0·25N-HNO_3 followed by 2 litres of N-aq. NH_3. Evaporate both eluates to dryness. Re-dissolve the acid fraction in 2N-HCl, adsorb on a smaller column, and elute with 0·25N-HNO_3, discarding the first 100 ml containing the bulk of the Fe. Evaporate to dryness, collect the residues in a NaCl matrix and examine spectrographically.

J. W. PRICE

1053. **Differential thermal analysis of kaolinite-illite mixtures.** R. J. W. McLaughlin (Dept. of Geology, Univ. Melbourne, Australia). *Trans. Brit. Ceram. Soc.*, 1960, **59** (5), 178–187.—The variable effects of the intimacy of mixing, of the concn. of illite on the 500° endothermic peak of kaolinite and of the products of reaction between the sample and the nickel sheath and thermocouple on the 850° peaks of illite (exothermic peaks above 900° are similarly affected) render differential thermal analysis a very unreliable quantitative method. J. A. SUGDEN

1054. **Analysis of enamels containing zirconium, antimony or cobalt.** J. Debras-Guédon, I. A. Voinovitch and M. Lancelot (Soc. Franç. de Céram., Paris). *Chim. Anal.*, 1960, **42** (9), 437–447.—Three schemes of analysis are outlined for enamels based on Sb, Zr and Co, respectively. Details are given for the determination of Pb, Si, Sb, Sn, Fe, V, Ti, Al, Ca, Mg, Zn, Mn, Co, Ba, Zr, Li, Na, K and B by gravimetric, volumetric and colorimetric methods. An appendix describes the simultaneous determination of Ti and V by spectrophotometry, the extinctions of the peroxide complexes being read at 400 and 450 mμ, and substituted in two simultaneous equations. J. H. WATON

1055. **Systematic separation of fission products by a solvent extraction method.** Toshiyasu Kiba, Shigeru Ohashi and Toyoo Maeda (Fac. Sci., Kanazawa Univ.). *Bull. Chem. Soc. Japan*, 1960, **33** (6), 818–821.—Radioactive nuclides (^{95}Zr, ^{95}Nb, ^{144}Ce, ^{147}Pm, ^{90}Y, ^{90}Sr, ^{106}Ru and ^{137}Cs) can be separated by four successive extractions with organic solvents by using 0·05 to 0·1 μC of each nuclide. Mixtures are shaken in separating-funnels for 5 to 10 min. and aliquots of the phases are evaporated or ignited before measuring the β-activity with an end-window Geiger-Müller counter. Nuclides in the organic phases can be extracted by shaking with HNO_3. *Procedure*—(*i*) Extract ^{95}Zr plus ^{95}Nb from 5M-HCl into $CHCl_3$ with cupferron; (*ii*) extract ^{144}Ce, ^{147}Pm and ^{90}Y from acetate buffer soln. (pH 4 to 5) into $CHCl_3$ with cupferron; (*iii*) extract ^{90}Sr from acetate buffer soln. (pH 8) into isobutyl methyl ketone with 2-thenoyltrifluoroacetone; (*iv*) extract ^{106}Ru from 5M-HCl with tributyl phosphate-kerosine (1:2) in the presence of $SnCl_2$ or into tributyl phosphate in the presence of hydroxylamine; ^{137}Cs remains in the final aq. soln. J. W. PRICE

See also Abstracts—**896**, Automatic phosphate and silica analysis. **898**, Spectrographic analysis of Ge, As, Se, Br, Sn, Sb and Te in powders. Spectrographic analysis of uranium oxides. Reciprocal effects in flame excitation of brasses. Oxygen and N in titanium. Influence of EDTA on flame-emission errors of Ca and Sr. Flame spectrophotometry of alkali metals. Oxygen in gases. Halogens in soln. Use of emission and mass spectrometry for high-purity metals. Oxygen in CO_2 by mass spectrometry. X-ray emission spectrography for metals. **908**, **909**, Indicators for Ca and Mg. **910**, Fluorochromic indicator for metals. **911**, Haematoxylin as indicator for metals. **917**, Determination of elements by the electron probe. **1111**, Trace, V, Fe and Ni in oils. **1112**, Trace V in oils. **1113**, S in coal. **1141**, V in biological materials. **1142**, W in biological materials. **1190**, Determination of K. **1222**, Ferrocyanide determination. **1329**, Determination of Pb and CN^-.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather and explosives.

1056. **The identification of organic substances by controlled pyrolysis and the application of gas chromatography. I. Fundamentals and analytical technique.** J. Janák (Gas Analysis Lab., Czech. Acad. Sci., Brno). *Coll. Czech. Chem. Commun.*, 1960, **25** (7), 1780–1789 (in German).—The sample of non-volatile or thermally unstable material is transferred to a platinum spiral, which is then heated electrically, and the pyrolysis products are examined with a gas chromatograph. The relative positions and heights of the several peaks recorded are characteristic of the substance examined and are reproducible under the same controlled conditions. Chromatograms are given for olive oil, albumin, atropine sulphate, phenylalanine and barbiturates. T. R. ANDREW

1057. **Ultra-violet fluorescence for detection of organic compounds on paper.** E. S. Rorem (Western Regional Res. Lab., Albany, Calif., U.S.A.). *J. Chromatography*, 1960, **4** (2), 162–165 (in English).—Solutions of quinine sulphate dihydrate (0·5 and 1%) in ethanol and of sulphosalicylic acid (1%) in acetone were used as dips for the detection of a wide variety of organic compounds on developed and dried chromatograms. After being dipped, the papers are dried at room temp. and viewed under u.v. light, and the spots become visible against the fluorescent background. Inorganic and non-volatile solvents interfere. The sapogenin lucernic acid has been detected on chromatograms for the first time. Minimum detectable amounts are tabulated for sugars, amino-acids and saponins.

S. M. MARSH

1058. **Application of polarography in organic chemistry.** H. W. Nürnberg (Max Planck Inst. f. Eisenforsch., Düsseldorf, Germany). *Angew. Chem.*, 1960, **72** (13), 433–449.—Apparatus and methods

are described, and the applications of polarography to constitutional and structural problems are discussed.

1059. **New chemical method for the determination of the water content of organic solvents.** K. F. Jahr and J. Fuchs (Inst. für Anorg. Chem., Freien Univ., Berlin-Dahlem, Germany). *Z. anal. Chem.*, 1960, **176** (4), 269–282.—The method described depends on the hydrolysis by water of t-butyl orthovanadate in the presence of an anhyd. satd. soln. of NH_3 in $CHCl_3$ or (for acetone) acridine in anhyd. light petroleum. An excess of base and of ester is added, resulting in the stoicheiometric hydrolysis of the ester to the insoluble vanadate, which can then be filtered off, washed with anhyd. ether, dissolved in $2N-H_2SO_4$, treated with H_3PO_4-H_2SO_4 soln., and titrated with $(NH_4)_2SO_4.FeSO_4$, with *N*-methyldiphenylamine-*p*-sulphonic acid (sodium salt) as indicator. E. G. CUMMINS

1060. **Oxidation processes in elementary analysis. IV. Influence of length of oxidation layer and speed of flow in combustion analysis.** G. Kainz and H. Horwatitsch (Anal. Inst., Univ. Wien, Austria). *Z. anal. Chem.*, 1960, **176** (3), 175–185 (in German). —It is shown that the oxidation efficiency of a tube-filling depends on four factors, *viz*, oxygen partial pressure of the oxide, effective CuO concn., reaction velocity (temp.) and time of contact. Equations for calculating these factors are given and discussed, with methane as test sample and CO_2 as carrier gas. B. B. BAUMINGER

1061. **Organic quantitative analysis. XXV. Investigation of the surface absorption of oxides of nitrogen by manganese dioxide during carbon and hydrogen determinations.** M. Večeřa and D. Šnobl (Forschungsinst. f. org. Synthesen, Pardubice-Rybitví, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2013–2021.—Manganese dioxide combines with nitrogen dioxide in a quantity proportional to its surface hydroxyl groups. On the basis of this finding, hydrated $MnO(OH)_2$ gel was found most advantageous for this absorption. Methods of preparation of the gel are described and details for its use in elementary analyses are given. B. HEINING

1062. **Determination of chlorine by the oxygen flask combustion method. A single unit for electrical ignition by remote control and potentiometric titration.** J. Haslam, J. B. Hamilton and D. C. M. Squirrell (I.C.I. Ltd., Plastics Div., Welwyn Garden City, Herts., England). *Analyst*, 1960, **85**, 556–560. —The combustion flask, firing adapter and titration electrodes are described diagrammatically; the automatic titrimeter has been described previously (Haslam *et al.*, *J. Appl. Chem.*, 1959, **9**, 65; 1960, **10**, 93). The sample ($\not> 30$ mg $\equiv$ 5 to 6 mg of Cl) is wrapped in filter-paper provided with a cotton-wool wick. The combustion vessel contains 1 ml of N-NaOH, 3 drops of 35% $NaHSO_3$ soln. and 3 ml of water, and is filled with oxygen. The adapter containing the sample is inserted and the wick is fired by remote control. After combustion, the adapter is disconnected, the sample holder is washed into the flask and the sides of the flask are rinsed to give a vol. of $\simeq 35$ ml. Excess of $NaHSO_3$ is destroyed with 100-vol. H_2O_2 (6 drops), and 0·15 ml of $N-HNO_3$ is added in excess of neutrality to methyl red. The electrode assembly is fitted and the magnetically stirred liquid is titrated with $0{\cdot}01N$-$AgNO_3$, the end-point being the point of max. deflection in the titration graph. A blank determination is made, and the $AgNO_3$ soln. is standardised under the same conditions by including NaCl in the absorption liquid in a blank combustion. Determinations with compounds of known Cl content indicated satisfactory accuracy. A. O. JONES

1063. **Detection of boron in organic compounds.** R. Neu (Chem. Forschungslab., Fa. Dr. Willmar Schwabe G.m.b.H., Karlsruhe-Durlach, Germany). *Z. anal. Chem.*, 1960, **176** (5), 343–346.—Reagent-grade diphenylcarbazone, as used for the colorimetric determination of metals, is used in the direct detection of B. The nineteen samples listed include seven benzeneboronic acids, all of which gave maximum extinctions at 551 mμ. E. G. CUMMINS

1064. **Problems of analytical determination of the carbonyl group.** B. Buděšínský (Res. Inst. Pharm. and Biochem., Prague). *Chem. Listy*, 1960, **54** (9), 916–946.—A survey and evaluation of various chemical and physico-chemical methods are given, with 295 references. J. ZÝKA

1065. **Gas-chromatographic determination of C_6, C_7 and C_8 olefins according to their carbon structures.** K. H. Nelson, W. J. Hines, M. D. Grimes and D. E. Smith (Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1110–1114.—The method is based on determining the amounts of the individual paraffins and naphthenes present in the sample before and after hydrogenation. The differences between the two analyses represent the olefins having the carbon structures of the corresponding paraffins and naphthenes. In this manner all the olefins having a particular carbon structure are reported as a sum. Two columns are used, one, packed with Chromosorb impregnated with silicone oil, to determine the hexanes, and the other, impregnated with squalane, to determine the heptanes and octanes. The accuracy of the method is within about $\pm 6\%$ of the amount present, based on the analysis of synthetic blends. G. P. COOK

1066. **Determination of vinylacetylene.** J. Vítovec and M. Šádek (Res. Inst. Synth. Rubber, Gottwaldov, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (7), 1972–1974 (in German).—An ampoule containing the sample ($\equiv 0{\cdot}01$ to 0·2 g of vinylacetylene) is dropped into a flask containing 50 ml of $0{\cdot}01N$-$AgNO_3$ and sufficient aq. NH_3 to give a clear soln., and 2 ml in excess. The flask is stoppered, the ampoule is crushed, and the mixture is stirred vigorously for 5 min. The ppt. of Ag vinylacetylide is filtered off and washed with water, and the excess of Ag is determined in the filtrate. By using a soln. of $AgNO_3$ in 20% ethanol the method has been applied to the determination of vinylacetylene (2 to 14%) in xylene with a relative error of $\simeq 0{\cdot}5\%$. T. R. ANDREW

1067. **Determination of butadiene by a chromatothermographic method.** V. A. Kolobikhin. *Zavod. Lab.*, 1960, **26** (7), 814–815.—A 45-cm column of Al_2O_3 is heated by means of a stationary furnace. The sample (18 to 20 ml) of butene - butadiene mixture is introduced into the air-free column and a current of CO_2 is passed at a rate of 50 ml per min. The temp. is gradually increased. The gases pass to a measuring burette where CO_2 is absorbed in KOH. At 70° butane appears, followed by butene at 90° and butadiene at 110°. G. S. SMITH

1068. **The use of S-alkyl-N-phenylthiuronium picrates, styphnates and picrolonates for the characterisation of alkyl halides.** J. Thomas and W. A. Baker (Dept. of Pharm., Univ., Manchester). *J. Pharm. Pharmacol.*, 1960, **12** (8), 460–465.—These derivatives have been prepared with 23 different alkyl groups. They melt sharply and may be used for the identification of all the lower primary and secondary alkyl halides studied. *Procedure*—Dissolve *N*-phenylthiourea (1 g) and the alkyl halide (1 g) in 50% ethanol (10 ml), heat under reflux for 1 hr. and divide into three portions. The reaction with alkyl chlorides is slow, but can be accelerated by the addition of NaI. Pour one portion into a satd. aq. soln. of picric acid (25 ml) and set aside for 30 min. To the second and third portions add styphnic acid (0·3 g) and picrolonic acid (0·3 g), respectively, heat to boiling, add enough 50% ethanol to dissolve the solid and allow to cool. Re-crystallise from 50% ethanol.
A. R. Rogers

1069. **Physical method of trace analysis after concentration by preparative gas chromatography. Determination of dichloroethylenes in carbon tetrachloride.** J. Serpinet (Soc. d'Électrochim., d'Électrométall. et des Aciéries Électriques d'Ugine, Pierrebenite, Rhône, France). *Chim. Anal.*, 1960, **42** (9), 433–436.—Trace amounts of substances are concentrated on the preparative-scale column already described (*Anal. Abstr.*, 1960, **7**, 569) and are then determined in the mass spectrograph. A gas chosen to come off the column at the same time as that being determined is added as tracer, carrier and internal standard. The procedure shows a precision of $\simeq$10%. The determination of $\simeq$1 p.p.m. of dichloroethylene in CCl_4 is described. Light petroleum (2%) is added, in which the hexane isomers act as tracers for *cis*- and *trans*-dichloroethylene, and H is used to elute the column. In the mass spectrograph, the ratio of the mass-96 dichloroethylene to the mass-71 internal standard is measured. The advantages and disadvantages of the method are discussed. J. H. Waton

1070. **Analytical control of the production of ethylene oxide by a direct oxidation of ethylene.** J. Janda (Res. Inst. Petrochem., Nováky, Czechoslovakia). *Chem. Zvesti*, 1960, **14** (7), 521–524.—The results of the measurements of the content of ethylene and O or ethylene and CO_2 during the control of the oxidation of ethylene to ethylene oxide by the use of continuous gas-analysers were improved by calculating the results from the volume changes during the reaction. J. Zýka

1071. **Determination of basic α-epoxides.** J. B. Leary (Anal. Devel. Dept., Eli Lilly & Co., Indianapolis, Ind.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (9), 606–608.—Mix the aq. soln., containing $\simeq$1 milli-equiv. of epoxide, with 0·25N-acetic acid (5 ml), add 0·1N-$Na_2S_2O_3$ (25 ml), heat at 70° for 15 min., cool in ice and immediately titrate with 0·1N-iodine, with starch as indicator. The coeff. of variation, calculated from 10 replicates, is ±1·3%.
A. R. Rogers

1072. **Polarographic study of organic peroxides.** E. J. Kuta and F. W. Quackenbush (Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1069–1072.—Polarograms were obtained, with an improved polarographic procedure, for 22 commercial organic peroxides having the following functional peroxide groups—hydroperoxides, peroxy-acids, peroxy-esters, six-membered bicyclic peroxides, diacyl peroxides and ketone peroxides. Twenty of the compounds showed one or more characteristic waves and were placed in five groups based on their $E_{\frac{1}{2}}$ values; two of the compounds failed to give waves under the conditions used (0·0 to −2·0 V). A linear relationship existed between diffusion current and concn. for all but two of the compounds. G. P. Cook

1073. **Identification of organic compounds. XXXVII. Paper-chromatographic separation of alcohols as xanthates.** J. Gasparič and J. Borecký (Forschungsinst. f. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *J. Chromatography*, 1960, **4** (2), 138–143 (in German).—The alkylxanthates of a series of alcohols were chromatographed on paper in systems of varied alkalinity. The xanthates were fixed by spraying with 10% nickel sulphate soln. and with ammoniacal silver nitrate soln. [5% $AgNO_3$ soln. - conc. aq. NH_3 (9:1)]. In homologous series the R_F value increases with the length of the alkyl chain from C_1 to C_{12}. In order to prevent decomposition of the xanthates, the medium must be sufficiently alkaline. Results are discussed and compared with those of previous work. The advantages and disadvantages of xanthates as derivatives for the paper-chromatographic separation of alcohols are enumerated.
S. M. Marsh

1074. **Refractometric determination of glycerol in crude glycerol with ion exchangers.** A. Kopecký, V. Klozar and E. Krejcar (Res. Inst. Fat Industry, Prague). *Průmysl Potravin*, 1960, **11** (8), 438–439.—The sample (1·8 to 2 g) is diluted with H_2O (5 to 6 ml) and the impurities are removed by passage through a column filled with a mixture of strong acid and strong basic ion-exchanger (Wofatit KPS 200 - Wofatit L 165) (1:1·5) (4 to 5 ml per min.). The percolate is diluted to 25 ml by washing the column with H_2O and glycerol is determined refractometrically. J. Zýka

1075. **Quantitative estimation of carbohydrates by paper partition chromatography.** S. B. Misra and V. K. Mohan Rao (with S. K. Bose) (Central Drug Res. Inst., Lucknow, India). *J. Sci. Ind. Res., India, C*, 1960, **19** (7), 173–176.—The intensity of colour developed by spraying paper chromatograms of D-glucose, D-galactose, D-glucuronic acid, D-arabinose and L-rhamnose with aniline hydrogen phthalate reagent (Partridge, *Nature*, 1949, **164**, 443) and heating and extracting into various solvents was investigated. The nature of subject carbohydrate, original solvent for running the chromatogram, solvent for eluting the coloured compound and the amount of water it contains, the time of running the chromatogram, the time and temp. of eluting the spots and the time of heating to develop the colour all affect the extinction and were investigated. As a result, a procedure was developed enabling these five carbohydrates to be determined to within ±6% for 10 to 100 μg. *Procedure*—The chromatograms of sample and standard must be run under identical conditions. The sprayed chromatogram is heated for 15 min. at 105° and the spots are eluted for 30 min. with 50 to 80% aq. acetic acid, acetone, ethanol or methanol, and the extinction is determined. E. J. H. Birch

1076. **Gas - liquid partition chromatography of methylated sugars.** H. W. Kircher (College of Agric., Univ. Ariz., Tucson, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1103–1106.—A mixture of the fully

methylated methyl α- and β-pyranosides of D-arabinose, D-xylose, D-glucose, D-mannose and D-galactose is resolved into 8 distinct peaks on a 6-ft. column of methylated hydroxyethylcellulose. The methyl glycosides of di- and tri-*O*-methylhexoses also pass through the column. The applicability of the method is demonstrated with starch, cellulose, guar gum and dextran. G. P. Cook

1077. **Analysis of 1,2-glycols and polyhydric compounds. V. Determination of glyceraldehyde.** L. Maros, I. Perl and E. Schulek (Inst. for Inorg. and Anal. Chem., L. Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1960, **66** (8), 319–321.—Two volumetric methods are described. In the first, glyceraldehyde is determined by direct iodimetric - sulphite titration. The second method involves oxidation of glyceraldehyde with periodic acid (45 g of $Na_3H_2IO_6$ in 500 ml of 3% H_2SO_4) and determination of the formaldehyde so formed by the bisulphite - cyanide method. Full working details are given and the methods are compared. The accuracy of each method is stated to be within ±5%. B. Vaszko

1078. **New methods of detection of glyoxal.** E. Sawicki and W. Elbert (Robert A. Taft Sanitary Engng Center, U.S. Dept. of Health, Education and Welfare, Cincinnati, Ohio). *Talanta*, 1960, **5** (1), 63–65.—Spot tests for glyoxal with identification limits down to 0·008 μg are described. A drop of sample and a drop of reagent are spotted on to paper and the spot is heated in steam. The reagent is a soln. in aq. HCl of one of the following compounds—1,2-dianilinoethane, 2,3-diaminonaphthalene, 2-aminothiophenol or 2-hydrazinobenzothiazole. The first 3 reagents give blue, green and blue colours, respectively, and the last gives a yellow fluorescence under u.v. light. G. Burger

1079. **Determination of [aromatic] glyoxals and their sodium bisulphite addition products by potentiometric titration.** J. G. Baldinus and I. Rothberg (Smith Kline & French Lab., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1176–1178. —The method is based on the Cannizzaro reaction. The glyoxals are converted into the corresponding mandelic acids and the alkali consumed is measured by potentiometric titration with acid. Because $NaHSO_3$ reacts with NaOH to form Na_2SO_3, the method can be extended to the bisulphite addition products, but, to obtain a satisfactory end-point, H_2O_2 must be added just before the end-point to oxidise the sulphite to sulphate. The standard deviation is ≃ ±0·55% from 10 determinations. G. P. Cook

1080. **Tetraphenylstibonium sulphate as a reagent for the qualitative analysis of organic acids.** H. E. Affsprung and H. E. May (Univ. Okla., Norman, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1164–1166.—The methods of preparation of the reagent and some organic acid derivatives are described. The derivatives are the salts of the tetraphenylstibonium ion and are prepared by adding aq. 0·05M-tetraphenylstibonium sulphate to aq. soln. of the acids. The salts are re-crystallised from n-hexane, H_2O or aq. ethanol; the m.p. are sufficiently sharp and far apart for easy differentiation of many acids. G. P. Cook

1081. **Determination of fatty acids of lower molecular weight by gas chromatography.** M. L. Vorbeck, L. R. Mattick, F. A. Lee and C. S. Pederson (New York State Agric. Exp. Sta., Cornell Univ., U.S.A.). *Nature*, 1960, **187**, 689.—A mixture of fatty acids was converted into methyl esters by reaction with diazomethane and injected into the gas-chromatographic column, which was operated at 92°, with helium as the carrier gas, flowing at 40 ml per min. The separation takes about 28 min. The same column is used for higher fatty acids by raising the temperature to 216° and increasing the flow rate to 60 ml per min. The relation between amounts of lower- and higher-molecular-weight fatty acids is found by comparison of the peaks of an ester that appears on each chromatogram. A. C. R. Hartley

1082. **Quantitative analysis of some C_{10} dibasic acids and associated monobasic acids by high-temperature gas chromatography.** R. C. Bartsch, F. D. Miller and F. M. Trent (U.S. Industrial Chem. Co., Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1101–1103.—Samples were quant. converted into methyl esters with diazomethane and the esters resolved at 236° on a 12-ft. column of specially prepared high-vacuum silicone grease or on a 4-ft. column of a polyester. Three aliphatic C_{10} dibasic and two C_9 monobasic acids were successfully resolved, recoveries of >98·5% being achieved for complex mixtures. The error was ±0·4% absolute. G. P. Cook

1083. **Titrimetric determination of carboxylic acid chloride.** L. J. Lohr (Explosives Dept., E. I. du Pont de Nemours & Co., Inc., Gibbstown, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1166–1168.—Aromatic and aliphatic carboxylic acid chlorides in tetrahydrofuran soln. can be directly titrated with a soln. of cyclohexylamine in tetrahydrofuran. The end-point is shown by a sudden change in the potential, measured with glass - calomel electrodes. Free HCl interferes and a correction must be made. The absolute accuracy and precision are within ±1%. G. P. Cook

1084. **Paper-chromatographic identification of cyanoacetic acid.** M. R. Subbaram (Nutrition Res. Lab., Indian Council of Med. Res., Hyderabad). *J. Sci. Ind. Res., India, C*, 1960, **19** (6), 156–157.—Cyanoacetic acid, a metabolic product of β-aminopropionitrile, can be developed on chromatograms with butanol - acetic acid - water (4:4:1) and determined in amounts of not less than 2·5 μg from the colour formed by spraying successively, with intermediate drying, with (*a*) 1 g of sulphanilic acid in 10 ml of conc. HCl diluted to 100 ml, (*b*) 500 mg of $NaNO_2$ in 100 ml of water, and (*c*) 10% Na_2CO_3 solution. Solutions (*a*) and (*c*) are mixed with an equal vol. of ethanol for spraying. This prevents spreading of the spots. J. I. M. Jones

1085. **Decomposition of short-chain dicarboxylic acid esters during separation on polyester gas-chromatography media.** R. G. Ackman, M. A. Bannerman and F. A. Vandenheuvel (Fisheries Res. Board of Canada, Halifax, Novia Scotia). *Anal. Chem.*, 1960, **32** (9), 1209.—When the methyl esters of short-chain dicarboxylic acids are analysed by gas - liquid chromatography with a polyester as the partition medium the two lowest members fail to give quant. results. Investigation showed that the proportion of dimethyl malonate declined rapidly with increase in temp. of the column. The decomposition is probably caused by the reaction of the esters with the polyester of the column, liberating the alcohol and the acid, the acid then being thermally decomposed to give CO_2 and a monobasic acid. G. P. Cook

1086. **Infra-red spectra of esters. I. Methyl, ethyl, n- and iso-propyl, and n-, iso- and s-butyl esters.** A. R. Katritzky, J. M. Lagowski and J. A. T. Beard (Univ. Chem. Lab., Cambridge, England). *Spectrochim. Acta*, 1960, **16** (8), 954–963.—Bands characteristic of the alkyl groups in 29 methyl, 27 ethyl, 13 n-propyl, 7 isopropyl, 13 n-butyl, 9 isobutyl and 6 s-butyl esters are tabulated and discussed. It has been found that each type of alkyl group shows a definite number of bands, the positions and intensities of which are reasonably constant. Assignments to specific molecular vibrational modes are suggested for most of the bands.

II. Acetates, propionates, n-butyrates, β-arylpropionates, acrylates, methacrylates, crotonates, β-arylacrylates, arylcarboxylates, formates, chloroformates and carbonates. A. R. Katritzky, J. M. Lagowski and J. A. T. Beard. *Ibid.*, 1960, **16** (8), 964–978.—Bands characteristic of the acyloxy-groups in 104 esters of the types mentioned are tabulated and discussed. It has been found that the nature of the alkyl group has relatively little effect on either the position or the intensity of these bands. Assignments to specific molecular vibrational modes, some of which conflict with the conclusions of Wilmshurst (*cf. J. Mol. Spectrosc.*, 1957, **1**, 201), are suggested for most of these bands. In the work described, all bands with $\epsilon_A > 10$, where $\epsilon_A = (1/Cl) \log_{10} (I_0/I)$, C being the concn. in moles per litre and l the cell length in cm, have been reported. Those that could not be correlated with either the alkyl or the acyloxy-group (<1% of the total) are listed. Measurements were made in $CHCl_3$ soln. and the regions 2000 to 1240 and 1200 to 800 cm^{-1} were covered. E. G. CUMMINS

1087. **Paper chromatography of 2,4-dinitrophenyl derivatives of amines.** A. M. Asatoor (Dept. of Chem. Pathology, Postgrad. Med. School, London, England). *J. Chromatography*, 1960, **4** (2), 144–152 (in English).—A series of 21 2,4-dinitrophenyl derivatives of amines was chromatographed on various treated papers, *viz*, silicone-treated, liquid paraffin-treated, acetylated and benzoylated. Solvents used were the upper phases of a mixture of $CHCl_3$, methanol and water (5:5:3), and of $CHCl_3$, methanol, water and liquid paraffin (5:5:3:2) for paraffin-treated paper. Results show that, for routine work, chromatography on paraffin-treated paper is the simplest and most satisfactory procedure. Human urine was analysed by the method and shown to contain methylamine, dimethylamine, ethylamine, pyrrolidine and piperidine. S. M. MARSH

1088. **Detection and determination of ethylenediaminetetra-acetic acid (EDTA) and its derivatives in complex media.** Y. Lembrez, J. Storck and B. Terlain (Lab. de Chim., Centre de Transfusion-Réanimation de l'Armée, Clamart, France). *Ann. Pharm. Franç.*, 1960, **18** (5), 285–294.—Interfering substances in the determination of EDTA are removed in simple cases by the pptn. of other complexing anions with $BaCl_2$ in ammoniacal soln. (pH > 10), which also leads to pptn. of heavy metals as hydroxides. In more complex cases, removal of protein can be effected with trichloroacetic acid. With plasma or serum, 5 ml is treated with 1 ml of 60% trichoroacetic acid soln. and 4 ml of the centrifugate is treated with 1·9 ml of 12N-aq. NH_3 and 0·1 ml of 10% aq. $BaCl_2$ soln.; the mixture is re-centrifuged and the centrifugate is used for the determination. With wine or urine, removal of pigment is necessary and, after pptn. with Ba, the supernatant liquid (6 ml) is treated with 0·5 ml of 10N-H_2SO_4, 1 ml of 25% poly(vinylpyrrolidone) soln. and 7·5 ml of satd. aq. $(NH_4)_2SO_4$ and filtered. The most general purification can be effected by adjusting the EDTA soln. (0·01 μg to 10 mg per ml) to pH 2 with acetic acid or NaOH and passing it through a column (250 × 15 mm) of Permutit 50 (H^+ form). EDTA, other ampholytes, NH_4^+ and other cations are retained on the column, which is washed with 20 ml of water. The column is then washed with 200 ml of N-aq. NH_3 which elutes only EDTA, other ampholytes (such as amino-acids) and NH_4^+. The soln. is evaporated to dryness *in vacuo* and the residue is dissolved in the minimum quantity of water. Determination of EDTA in the purified soln. is effected volumetrically by adding to 51 ml of the test soln. (pH 3) 20 ml of 5% thiourea soln. in HNO_3 - glycine buffer (pH 1·6) and 5 ml of 0·005M-Bi^{3+}, and back-titrating with 0·005M-EDTA (disodium salt) until the colour is discharged. Alternatively, the Bi - thiourea complex is determined colorimetrically at 420 to 460 mμ. For less concentrated EDTA soln., the standard and test soln. (5 ml at pH 3) are treated with 2 ml of a soln. of Fe [8 ml of 0·01M-Fe^{3+}, 1 drop of 20-vol. H_2O_2 soln., and HCl - glycine buffer (pH 2·4) to 100 ml], 1·75 ml of isoamyl alcohol and 4 ml of NH_4SCN soln. (10 g in 10 ml of buffer at pH 2·4). After 5 min. the org. layer is removed and the extinction is measured. The precision is ±3% over the range 0·5 μg to 10 mg of EDTA per ml. E. J. H. BIRCH

1089. **Paper chromatography of some organo-tin compounds.** D. J. Williams and J. W. Price (Tin Res. Inst., Fraser Rd., Greenford, Middx., England). *Analyst*, 1960, **85**, 579–582.—Of the organo-tin compounds, R_nSnX_{4-n}, where R is an aryl or alkyl group and X an acid group, certain dialkyltin compounds (R_2SnX_2) are used as stabilisers for poly(vinyl chloride), and members of the group R_3SnX are likely to find increasing use as fungicides and bactericides. The paper chromatography of a number of organo-tin compounds is described and the R_F values are tabulated. The developing solvents are butanol - pyridine - water, butanol - ethanol - water, butanol - aq. NH_3 - water and aq. pyridine. The spraying reagent is ethanolic catechol violet soln. applied after exposure of the chromatogram to u.v. light to oxidise R_4Sn and R_3SnX. For quant. results, the relevant zones are cut out and wet-ashed, and the resulting inorganic Sn is determined turbidimetrically with 4-hydroxy-3-nitrobenzenearsonic acid. Diphenyltin and triphenyltin compounds can be recovered quantitatively. A. O. JONES

1090. **Determination of alkylbenzenes.** D. E. Nicholson and S. H. Hastings (Humble Oil & Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (9), 1212.—Infra-red data are given for isopropyl-, n-propyl-, 3-ethyl-1-methyl-, 4-ethyl-1-methyl-, 1,3,5-trimethyl-, 2-ethyl-1-methyl-, t-butyl-, 1,2,4-trimethyl- and 1,2,3-trimethyl-benzenes.

1091. **Determination of C_{10} alkylbenzenes.** S. H. Hastings and D. E. Nicholson (Humble Oil & Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (9), 1211.—Infra-red data are given for 4-ethyl-1,2-dimethyl-, 2-ethyl-1,3-dimethyl-, 3-ethyl-1,2-dimethyl-, 1,2,4,5- and 1,2,3,5-tetramethyl-benzenes.

1092. **Determination of C_{10} alkylbenzenes.** D. E. Nicholson and S. H. Hastings (Humble Oil & Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (9), 1212.—Infra-red data are given for 1,3-diethyl-, 1-methyl-3-n-propyl-, 1-methyl-4-n-propyl-, n-butyl- and 1,2-diethyl-benzenes.

1093. **Determination of C_{10} and C_{11} alkylbenzenes.** D. E. Nicholson and S. H. Hastings (Humble Oil & Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (9), 1211.—Infra-red data are given for 2-ethyl-1,4-dimethyl-, 4-ethyl-1,3-dimethyl-, 1-methyl-3-t-butyl- and 4-ethyl-1,2-dimethyl-benzenes.

1094. **Interaction of quinones and hydrocarbons investigated by microscopic mixed-fusion analysis.** D. E. Laskowski (Armour Res. Foundation of Illinois Inst. of Technol., Chicago, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1171–1176.—A study has been made of the tendency of non-halogenated quinones to form addition compounds with aromatic hydrocarbons and, of the various compounds investigated, 2,5-diphenyl-1,4-benzoquinone seems to be the most promising as a sub-classification reagent for use in mixed-fusion analysis. K. A. PROCTOR

1095. **Routine analysis of phenols by gas - liquid chromatography.** D. S. Payn (North Thames Gas Board, Beckton, East Ham, London, England). *Chem. & Ind.*, 1960, (34), 1090.—A copper column (8 ft. × 4 mm) packed with 15% w/w of di-n-octyl sebacate on 60 to 100-mesh "Embacel" is used at 178° with H at 25 to 30 ml per min. and a flame detector. The sample (1 to 2 μl) is injected on to the column and the time for complete elution is $\simeq$40 min.; the column must be re-packed after 150 hr. Relative retention times are, for phenol 1·00; *o*-cresol 1·24; 2,6-xylenol 1·43; *m*- and *p*-cresols 1·49; *o*-ethylphenol 1·75; 2,4- and 2,5-xylenols 1·84; 2,3-xylenol and *p*-ethylphenol 2·20; 3,5-xylenol and *m*-ethylphenol 2·26; and 3,4-xylenol 2·55. R. M. S. HALL

1096. **Amperometric titration of some organic reducing reagents with potassium dichromate.** N. S. Sukhobokova. *Vestn. Leningrad Univ.*, 1960, No. 16, *Ser. Fiz. i Khim.*, (3), 149–150.—Quinol, *p*-methylaminophenol, *p*-aminophenol and *p*-phenylenediamine are determined by amperometric titration at 21° with 0·2N-$K_2Cr_2O_7$ in 0·5 to 2N-H_2SO_4, with either a platinum-wire micro-electrode (2 to 2 mm long fused into a glass tube) or a platinum-disc electrode (diameter 5 mm, mounted in a glass tube) at +1 V *vs.* the S.C.E. The electrodes rotate at 800 r.p.m. Concn. are determined with an accuracy within 6% and there is good reproducibility of titration curves. K. R. COOK

1097. **Polarographic determination of terephthalic acid in mixtures of phthalic acid isomers.** M. E. Hall and R. C. McNutt (Chemstrand Corp., Decatur, Ala., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1073–1074.—Terephthalic acid can be determined at concn. as low as 0·1% in mixtures of phthalic acid isomers. The supporting electrolyte is M-LiOH and the $E_{\frac{1}{2}}$ value for the first wave, which is used for quant. measurement, is $\simeq -1{\cdot}93$ V *vs.* the S.C.E. The diffusion current is linear with concn. for up to 30 mg per 25 ml of electrolyte, and the repeatability at the 2·0% level for triplicate determinations is ±0·1%. G. P. COOK

1098. **Polarographic determination of nitrocyclohexane.** Ya. I. Tur'yan, Yu. M. Tyurin, P. M. Zaĭtsev and E. A. Karavaeva. *Zavod. Lab.*, 1960, **26** (7), 810–813.—In acid (pH $\not<$ 1·2) and neutral soln. nitrocyclohexane gives a well-defined polarographic wave. At pH > 4 a second wave appears. At pH $\not>$ 4 the $E_{\frac{1}{2}}$ ($\simeq -0{\cdot}55$ V *vs.* the S.C.E.) scarcely changes with concn. At pH 6·3 the $E_{\frac{1}{2}}$ is between $-0{\cdot}73$ (2×10^{-3}M) and $-0{\cdot}77$ ($1{\cdot}36 \times 10^{-5}$M). G. S. SMITH

1099. **Determination of *m*-toluidine in *N*-ethyl-*m*-toluidine.** K. B. Whetsel, W. E. Roberson and M. W. Krell (Tennessee Eastman Co., Kingsport, Tenn.). *Anal. Chem.*, 1960, **32** (9), 1212.—Infra-red data are given.

1100. **Infra-red analysis of phenylchlorosilanes in the caesium bromide region.** M. E. Grenoble and P. J. Launer (Silicone Products Dept., General Electric Co., Waterford, N.Y., U.S.A.). *Appl. Spectroscopy*, 1960, **14** (4), 85–90.—The i.r. spectra of seven phenylchlorosilanes have been measured over the range 2 to 30 μ. The range 2 to 15 μ, in which the spectra are very similar, was recorded with a Perkin-Elmer model 21 spectrophotometer with a sodium chloride prism, and the region 15 to 30 μ with a Perkin-Elmer model 321 double-beam double-pass spectrometer and a caesium bromide prism. The latter instrument was flushed with N or dry air to eliminate interference by water-vapour absorption, and in this region marked differences between the compounds were recorded and attributed to Si–Cl stretching and aromatic-ring deformation vibrations. The region was also recorded successfully on a Perkin-Elmer KBr Infracord. Quantitative analyses of mixtures of diphenyldichlorosilane (**I**), phenyltrichlorosilane (**II**) and chlorobenzene were made and small amounts (0 to 5%) of **II** in **I** were determined with a mean error of ±0·1%. P. T. BEALE

1101. **Paper chromatography of aminonaphtholsulphonic acids.** Z. Gasparič, J. Kolšek and M. Perpar (Inst. Org. Chem., Univ. Ljubljana, Yugoslavia). *ChemikerZtg*, 1960, **84** (19), 635–636.—Good separation of aminonaphtholsulphonic acids was achieved with t-butanol - n-butanol - H_2O (4:3:3). After being developed for 10 to 12 hr. the chromatogram was dried and examined under u.v. light; most of the aminonaphtholsulphonic acids exhibit strong fluorescence. R_F values and other data are listed for 16 compounds. Reference standards were prepared from technical-grade materials by purifying them on columns of Al_2O_3 with 50% methanol soln. containing NH_3 at pH 9·2 to 10·0; the acids were then re-crystallised from HCl soln. Owing to the proximity of their R_F values, J- and γ-acids had to be separated by a modified procedure. G. P. COOK

1102. **The specificity of the modified Keller reaction.** H. P. Rieder and M. Böhmer (Psychiatr. Clinic, Univ., Basel, Switzerland). *Helv. Chim. Acta*, 1959, **42** (6), 1793–1798.—A modification of the Keller reaction is described and is shown to give characteristic absorption curves with indoles and pyrrole suitable for their determination. Hydroxyindoles and lysergic acid derivatives can be determined by this method. N. E.

1103. **Detection and determination of indole compounds on paper and in solution.** H. G. Leemann and H. Weller (Sandoz, Basel). *Helv.*

Chim. Acta, 1960, **43** (5), 1359–1364 (in German).—Exposure to HCl gas in the van Urk method for indole derivatives (*cf. Pharm. Weekbl.*, 1929, **66**, **473**) may be replaced by reaction with toluene-*p*-sulphonic acid to produce coloured complexes with absorption maxima characteristic of the derivative and useful for detection and determination. The sample (2 to 10 μg) is spotted on to filter-paper and developed with isopropyl alcohol - aq. NH_3 - water (8:1:1); the strip is drawn through a 20% methanol soln. of toluene-*p*-sulphonic acid and the extinction is measured after scanning for the maximum absorption band. Alternatively, 2 to 100 μg of the substance is dissolved in a 12% acetic acid soln. of toluene-*p*-sulphonic acid, the soln. is heated on an oil bath at 70° for 1 hr., and the extinction is measured. J. L. PROSSER

1104. **The qualitative and quantitative determination of pyridine derivatives by means of infra-red absorption spectroscopy.** I. Arendt and E. Asmus (Tech. Univ., Berlin-Charlottenburg 2, Germany). *Z. anal. Chem.*, 1960, **176** (5), 321–332.—Spectra are given, and the characteristic ring deformation and CH deformation bands in KBr discs and in dioxan and CS_2 (when the compounds are soluble therein) of 2-, 3- and 4-cyanopyridine, 6-cyano-2-methylpyridine, pyridine 2-, 3- and 4-aldoxime, 2-, 3- and 4-pyridyloxymethanesulphonic acid and 2,6-dicyanopyridine are tabulated. The analysis of binary and ternary mixtures is discussed. E. G. CUMMINS

1105. **Potentiometric derivative titration of melamine.** V. Kaláb and J. Řepiš (Chem. Závod. W. Piecka, Werk Zilina, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2237–2239 (in German).—The method is based on continuous titration of aq. melamine soln. with HCl, whereby the potential derivatives are directly registered. The apparatus consists of an automatic burette and stirrer, a calomel electrode and a platinum - iridium electrode. A non-electrolytic condenser (20 μF) and a sensitivity regulator are coupled between the electrodes. A mirror galvanometer (sensitivity 1×10^{-8} amp.), which is illuminated by a slit lamp, is coupled to the regulator. The reflected beam falls on to the slit of a drum (22·5 cm circumference), which contains photographic paper and revolves once in 6 min.; N-HCl flows continuously from the burette under the surface of the solution (1 ml per 20 sec.), and each 0·5 ml is recorded by pressing a key connected to the lamp illuminating the slit of the drum. The sample (1 g) is dissolved in 200 ml of H_2O by boiling. After cooling, the beaker is placed under the titration assembly and the stirrer is started. After 6 ml of HCl has been added the lamp and drum are switched on, the sensitivity reducer is set to the ratio 1:1 and the titration is continued to the maximum. When the beam returns to its original position the titration is stopped. The error of the method is $\pm$0·1%. A procedure is also described for the simultaneous determination of ammeline and melamine. B. HEINING

1106. **Direct determination of C3 to C5 hydrocarbons in olefinic and non-olefinic gasolines by gas - liquid chromatography.** D. D. Zakaib (British American Oil Co., Montreal, Canada). *Anal. Chem.*, 1960, **32** (9), 1107–1110.—C_3 to C_5 hydrocarbons are determined directly on a 16-ft. column packed with 4 ft. of di-(2-ethylhexyl) sebacate and 12 ft. of tri-*m*-tolyl phosphate saturated with silver nitrate, both on firebrick. The column temp. is 30° and the rate of flow of the carrier gas (He) is 60 ml per min. When all the light components have been eluted and their peak areas recorded, the flow of He through the column is reversed and the C_6 hydrocarbons are eluted and recorded in the same manner. The retention times for about 20 hydrocarbons are listed. An analysis of a petroleum of known composition gave results within $\pm$0·2% (absolute). G. P. COOK

1107. **Separation and infra-red spectroscopic determination of non-ionic octylphenoxyethanol additives in gasoline.** R. M. Sherwood and F. W. Chapman, jun. (Atlantic Refining Co., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1131–1132.—The sample is evaporated to low bulk on a steam bath and the residue, together with CCl_4, is passed through a column of activated alumina, which adsorbs the octylphenoxyethanol (**I**). The column is washed with CCl_4 and then with a 5% soln. of pyridine in CCl_4 to remove interferences. After elution with a 20% soln. of isopropyl alcohol in CCl_4, the **I** is determined from its i.r. absorption. The precision and accuracy are $\pm$3% (relative) at the 100 p.p.m. level of **I** in petroleum. G. P. COOK

1108. **Applications of Carbowax 400 in gas chromatography for extreme aromatic selectivity.** L. R. Durrett. Symposium on Gas Chromatography, 137th Meeting, American Chemical Society (April 1960).—A gas - liquid chromatography method has been developed for the determination of the aromatic content of aviation gasoline, taking advantage of the selective retention of aromatics by Carbowax 400. All paraffinic hydrocarbons in aviation gasoline are eluted prior to the elution of benzene. This method, which requires approx. 45 min., is superior to the silica-gel adsorption method (ASTM D936–55) and the fluorescent-indicator adsorption method (ASTM D1319–58T) currently used to determine the aromatic content of aviation gasoline. A further application is the determination of hydrocarbon impurities in high-purity petroleum benzene and toluene. FUEL ABSTR.

1109. **Volumetric technique utilising molecular sieves for determination of n-paraffin content of olefin-free petroleum distillates.** L. P. Larson and H. C. Becker (Texaco Research Center, Beacon, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1215–1216.—When a mixture of normal, branched-chain and cyclic hydrocarbons is adsorbed on a synthetic zeolite with an effective channel width of 5 Å there is a corresponding decrease in the vol. of the sample as the adsorption proceeds. This change in vol. may be used to determine the amount of normal paraffins in an olefin-free distillate. Only simple apparatus is required, and the method is applicable to a sample having a wide boiling-range. No interference due to surface adsorption is encountered. K. A. PROCTOR

1110. **Sensitive spot-test for nitrogen compounds in petroleum fractions.** P. V. Peurifoy and M. Nager (Shell Oil Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1135–1137.—The method is based on the visual comparison of coloured spots produced by the reaction of compounds containing N with tetracyanoethylene on filter-paper. The test can be applied either by simple spraying or by using the Weisz ring-oven technique. Colours for about

50 compounds are listed. Comparisons made against pure compounds as standards indicate that the average deviation from the true value is ± 20 p.p.m. in the 100 p.p.m. and $\pm 10\%$ in the 100 to 1000 p.p.m. range. The results also agreed reasonably well with those obtained by a modified Kjeldahl technique. G. P. Cook

1111. **Quantitative determination of traces of vanadium, iron and nickel in oils by X-ray spectrography.** C. W. Dwiggins, jun., and H. N. Dunning (Bureau of Mines, Petroleum Res. Center, U.S. Dept. of the Interior, Bartlesville, Okla.). *Anal. Chem.*, 1960, **32** (9), 1137–1141.—In the method described, V, Fe and Ni are quant. determined in oils by X-ray spectrography with a Co - Cr internal standard. When an internal standard is undesirable, an emission - absorption method is used. Advantages of these methods in comparison with several X-ray procedures are enumerated. K. A. Proctor

1112. **Colorimetric determination of the vanadium content of residual oils by 8-hydroxyquinoline.** R. J. Nadalin and W. B. Brozda (Westinghouse Res. Lab., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1141–1143.—The method is based on the extraction of vanadium 8-hydroxyquinolinate into $CHCl_3$ in the presence of Ca - EDTA at pH 5·5. The extinction of the extract is measured at 550 mμ. Only Sn, Ti and W interfere seriously. Beer's law is obeyed in the range 0 to 100 μg, and 2 μg of V can be detected. Oil samples are dry-ashed and the residue is dissolved in acid before determination of the V. Results from the analysis of seven oil samples showed good agreement with those obtained by the ASTM tungstophosphate method. G. P. Cook

1113. **Rapid and precise determination of total sulphur in coal.** A. C. Bhattacharyya and B. P. Bhaduri (Coal Survey Station, Raniganj, India). *J. Sci. Ind. Res., India, B*, 1960, **19** (6), 212–214.—Total S in coal is determined by the oxygen flask combustion method, with a platinum - rhodium gauze catalyst. Weigh 0·1 to 0·2 g of sample in a tube of fine-quality cigarette paper closed at one end by twisting. After weighing close the upper end by twisting, wrap inside a second cigarette paper and fold the packet in a platinum - rhodium gauze (2 in. $\times$ 1 in., 200 mesh) leaving part of the twisted paper protruding from the lower end as a wick and compressing the gauze to prevent the packet falling out. Suspend the gauze from a platinum hook on a glass rod through a rubber stopper to fit a 1-litre conical flask containing 10 ml of 20-vol. H_2O_2. Flush out the flask with O for 3 to 4 min., then light the paper wick of the sample and, after removing the O-inlet tube, insert the rubber stopper in the flask. The sample burns completely in 60 to 90 sec. When combustion is complete, cool the flask in running water and allow to stand for 30 min. Remove the stopper, wash the ash of the sample into the flask, add 5 ml of conc. HCl and boil for 10 min. Filter, neutralise the filtrate with aq. NH_3, with methyl red as indicator, acidify with 1 ml of conc. HCl, boil and precipitate SO_4^{2-} from the hot soln. with 5 ml of 10% $BaCl_2$ soln. and determine gravimetrically. Alternatively, determine the $BaSO_4$ with EDTA. Comparison with the standard Eschka procedure on 18 samples of coal containing 0·3 to 6·2% of S gave results that differed by $-0{\cdot}02$ to $+0{\cdot}06\%$, with a mean difference of $+0{\cdot}011\%$. J. I. M. Jones

1114. **Methods for the analysis and testing of coal and coke. Part 9: Phosphorus in coal and coke.** British Standards Institution (2 Park Street, London, W.1). B.S. 1016: Part 9: 1960. Amendment No. 1 (15.7.60).—Three corrigenda are included.

1115. **Determination of toluenesulphonates and xylenesulphonates in the presence of tetrapropylenebenzenesulphonates in liquid detergents.** E. Heinerth, H. van Raay and G. Schwarz (Anal. Lab. der Henkel-Werke, Düsseldorf). *Fette, Seif., Anstrichmitt.*, 1960, **62** (9), 825–826.—Dissolve the crude ethanolic extract (0·5 to 0·7 g) in H_2O (1 litre), mix a 20-ml aliquot with a satd. aq. soln. of NaCl (10 ml), 0·5N-NaOH (1 or 2 drops), 0·04% bromophenol blue soln. (4 drops) and $CHCl_3$ (15 ml) and titrate with 0·004M-benzylcetyldimethylammonium chloride with vigorous shaking. Under these conditions tetrapropylenebenzene passes into the $CHCl_3$ phase. Titrate another 20-ml aliquot with the same vol. of reagent in the absence of indicator, shake well, discard the $CHCl_3$ phase, remove $CHCl_3$ from the aq. phase by boiling, cool, dilute to 200 ml and measure the extinction at the absorption max. (near 220 mμ). A. R. Rogers

1116. **Determination of lower fatty acid esters in volatile oils. IV. Micro-analysis of volatile oils.** R. Pohloudek-Fabini and K. Luthardt (Pharm. Inst. der Ernst-Moritz-Arndt-Univ., Greifswald, Germany). *Pharm. Zentralh.*, 1960, **99** (8), 488–492.—The procedure described previously (*cf. Anal. Abstr.*, 1959, **6**, 2239) has been modified to allow the semi-quant. ($\pm 10\%$) determination of the esters in a 10-mg sample of oil. The spots of the Fe^{III} - hydroxamic acid complex are separately eluted from the paper chromatogram with absolute methanol (10 ml), and the soln. are measured in a Pulfrich photometer with filter S53 against a 0·4% methanolic $FeCl_3$ soln. as blank. Beer's law is obeyed for <10 μmoles of ester; ϵ is the same for all the lower esters. A. R. Rogers

1117. **Polarographic determination of thymol and carvacrol in *Thymus vulgaris* and *Thymus serpyllum* herbs and their volatile oils.** J. Frgala and J. Knap (Galena, Komárov u Opavy, Czechoslovakia). *Českosl. Farm.*, 1960, **9** (7), 342–345.—Thymol and carvacrol are converted into the polarographically active azo-derivatives by coupling with *p*-diazobenzenesulphonic acid. *Procedure for herbs*—To the sample (10 g) add H_2O (100 ml), mix, add tartaric acid (5 g) and NaCl (10 g) and distil with steam. Transfer an aliquot of the distillate into a 50-ml flask, neutralise with 0·2M-NaOH, add ethanol (10 ml) and a freshly prepared soln. of *p*-diazobenzenesulphonic acid and dilute to volume with a buffer soln. (pH 7·5). After 30 min. register the polarographic wave from $-0{\cdot}1$ V. Evaluate by the method of standard addition. *Procedure for volatile oils*—Dilute the sample (1 g) with light petroleum (30 ml) and extract successively with 30, 20, 20, 10 and 10 ml of 0·2M-NaOH. Dilute the extract with 0·2M-NaOH to 100 ml, neutralise an aliquot with conc. acetic acid to pH 7 to 8, and proceed as described above. J. Zýka

1118. **The application of $\Delta\epsilon$ analysis to pharmaceuticals: the determination of eugenol.** J. C. Demetrius, jun., and J. E. Sinsheimer (Coll. of Pharm., Univ. of Rhode Island, Kingston). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (8), 523–525.—To determine eugenol in clove oil or in eugenyl acetate, mix the sample (60 mg) with N-NaOH (3 ml)

and ethanol (15 ml) by shaking for 5 min., heat in a boiling-water bath for 15 min., with occasional shaking, cool and dilute to 250 ml with ethanol. To one 10-ml aliquot add N-NaOH (1 ml) and dilute to 100 ml with H_2O; to a second add 0·1N-H_2SO_4 (2 ml) and dilute to 100 ml. Measure the extinction of the alkaline soln. at 296 mμ relative to the acid soln., and subtract a correction for the extinction difference of a reagent blank. The results appear to be accurate to within $\simeq \pm 1\%$. Pharmaceutical preparations containing eugenol may be assayed similarly. A. R. Rogers

1119. **Identification of organic compounds. XXXV. Paper chromatography of Sudan dyes.** J. Gasparič and M. Matrka (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (7), 1969–1972 (in German).—The R_F values of 23 water-insol. azo-dyes have been determined on paper impregnated with dimethylformamide, liquid paraffin or lauryl alcohol, with cyclohexane, ethanol - water (4:1), ethanol - conc. aq. NH_3 (4:1), ethanol - water (1:1) and ethanol - conc. aq. NH_3 (1:1) as developing solvents. T. R. Andrew

1120. **Paper-chromatographic [wood] pulp analysis by relative concentration densitometry.** C. D. Warburton and L. Weisler (Eastman Kodak Co., Rochester, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1114–1117.—A direct densitometric technique, by which frequent calibration is eliminated, is described for the determination of the glucose to mannose to xylose ratio on paper chromatograms of pulp hydrolysates. Under the conditions described, a constant ratio is obtained at all concn. levels and no standard soln. are required. The method is rapid and simple, and useful for routine analyses. K. A. Proctor

1121. **Quantitative chemical analysis of binary mixtures of nylon 6 or nylon 6·6 and certain other fibres.** British Standards Institution (2 Park Street, London, W.1). B.S. 3069:1959. Amendment No. 1 (30.5.60).—The directions for preparing a test specimen have been revised.

1122. **Surface-active alkylene oxide condensation products. Determination of polyoxyethylene glycols in selected cationic ethylene oxide products.** J. V. Kilheffer, jun., and E. Jungermann (Armour Industrial Chemical Co., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1178–1180.—The polyglycols are isolated as dil. aq. soln. by batch treatment with ion-exchange resins, and the amount and the average mol. wt. of the compounds are measured by a spectrophotometric and a dichromate total oxidation procedure, respectively. Recoveries are 100% in the 12 to 37-milli-equiv. range. G. P. Cook

1123. **Applications and limitations of turbidimetric titration as a method of characterising solutions of polymers and mixed polymers.** P. E. M. Allen, R. Hardy, J. R. Majer and P. Molyneux (Univ. Birmingham, England). *Makromol. Chem.*, 1960, **39** (1–2), 52–66 (in English).—The results of a study of the turbidimetric titration of various polymers in several solvents with several precipitating agents are reported and discussed, with special reference to the two assumptions, basic to the turbidimetric procedure, that the turbidity produced by the slow addition of non-solvent to a stirred soln. of polymer can be related to the amount of polymer that has precipitated, and that the polymer precipitating at a certain ratio of solvent to non-solvent is identical with the discrete fraction that precipitates at that ratio. The procedure is described. It is shown that, for a mixture of polymers, the first assumption is valid for the first polymer to be precipitated, provided that the polymers are chemically distinct and have separate precipitation ranges; turbidities due to the pptn. of subsequent polymers or fractions may be affected by the presence of the suspension of the earlier polymer. The second assumption is not necessarily true for mixtures containing block or graft copolymers, since in some systems copolymer was pptd. together with the first polymer, and the most soluble polymer was pptd. together with the copolymer. It follows that the technique should only be used for analytical purposes when it can be conclusively shown, by means of preset or known mixtures, that the assumptions are in fact valid for the system. Electron micrographs of samples taken during titrations of soln. of single polymers show that assumptions that the particles are uniform in size and of regular geometrical shape are not valid. R. E. E.

1124. **Determination of polymerisation accelerators by titration in non-aqueous medium.** V. N. Dmitrieva and N. P. Dzyuba (Kharkov Works of Dental Materials). *Zavod. Lab.*, 1960, **26** (7), 813–814.—To determine dimethylaniline or dimethyl-*p*-toluidine in methyl methacrylate, the sample (2 g) is dissolved in 10 ml of glacial acetic acid and titrated with 0·1N-$HClO_4$ in glacial acetic acid in the presence of two drops of a 0·1% soln. of crystal violet in glacial acetic acid. G. S. Smith

1125. **Analysis of copolymers of vinyl cyanide by infra-red spectrophotometry.** C. Gentilhomme, A. Piguet, J. Rosset and C. Eyraud (Battelle Mémorial Inst. de Genève). *Bull. Soc. Chim. France*, 1960, (5), 901–906.—The i.r. spectra of copolymers of vinyl cyanide with 2% and 5% of acrylic acid and with 5% of methyl methacrylate and 5% of methyl acrylate, respectively, are studied in the form of dried or undried powders suspended in liquid paraffin or of films formed by evaporation from dimethylformamide soln. In the case of the copolymers with acid examined by the film technique, the CO band at 1728 cm^{-1} has shoulders. The dry powder gives a CO band at 1738 cm^{-1} but this has shoulders if the powder is not dry. The methyl methacrylate copolymer (powder or film) gives a CO peak at 1727 cm^{-1} and methyl acrylate at 1733 cm^{-1}. The interference by water or traces of dimethylformamide with the acid spectrum is discussed. The i.r. spectra of model mixtures of isobutyronitrile with isobutyric acid are studied for comparison. It is concluded that polymers should be examined as dried powders and the CO group is recognised by the peaks mentioned. The acrylate and methacrylate structures are distinguished by skeletal vibrations giving peaks at 1170 cm^{-1} for the isopropyl structure and at 1125 cm^{-1} for the t-butyl structure. E. J. H. Birch

1126. **Spectroscopic analysis of poly(vinyl chloride) compounds. II. Emission spectrographic analysis.** W. J. Bennett and E. M. Law (Northern Electric Co. Ltd., Wire and Cable Div., Lachine, Que., Canada). *Appl. Spectroscopy*, 1960, **14** (4), 101–103.—A method is described for the analysis of stabilisers and fillers in P.V.C. compounds used in wire and cable insulations. The plasticiser is

extracted with ethyl ether, and the resin component containing the stabiliser and filler is dissolved in tetrachloroethane; the soln. is diluted with tetrahydrofuran and centrifuged; the metallic compounds separate as a dry powder. This is mixed with cupric oxide and graphite in the ratio 1:19:20 and compressed as a pellet on the end of a graphite electrode. A spark-like discharge from a Multi-source is used to excite the spectra of Pb, Si, Al, Ca and Sb. Coeff. of variation are $\simeq 2$ to 3%, and results of analyses of several samples showed good agreement with theoretical values. P. T. BEALE

1127. **Polarographic determination of metal driers in a non-aqueous solution.** E. J. Kuta (Inst. Marine Resources, State Univ., Berkeley, Calif., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1065–1068—Concn. from 10^{-4} to $1{\cdot}5 \times 10^{-3}$M of a Co, Cu, Fe, Pb, Mn, Ni or Zn drier (as octoate) can be rapidly and accurately determined by dissolution of the sample (0·025 to 0·05 g) in benzene - methanol (1:1) containing 0·3M-Na acetate, followed by polarography of the soln. (50 ml) under N between 0 and 2 V *vs.* the mercury-pool anode. The wave height is corrected for the Na acetate soln., and the concn. of drier, which is directly proportional to the diffusion current, is obtained from the calibration curve. The procedure is applicable to p.p.m. of drier either singly or in mixtures. Rare-earth elements, Ca and Zr do not interfere. The presence of free fatty acids and peroxides prevents the method being applied to the determination of Fe in menhaden oil. W. J. BAKER

1128. **Determination of the acid and saponification values of waxes with fluorescent indicators.** W. Hessler and H. Marsen. *Fette, Seif., Anstrichmitt.*, 1960, **62** (7), 579–584.—Dark-coloured waxes make the ordinary titrations to phenolphthalein or alkali blue impossible. A method (and the apparatus required) is described in which the wax is dissolved in xylene - ethanol (1:1) and fluorescent indicators (dissolved in ethanol) are used. Titration curves with 4 different indicators and results for commercially obtainable waxes are given. The use of β-methylumbelliferone is preferred. I. DICKINSON

1129. **Analysis of products of chloroprene production by two-step gas chromatography.** J. Brodský, M. Mačka and O. Mikl (Res. Inst. Synth. Rubber, Gottwaldov, Czechoslovakia). *Chem. Průmysl*, 1960, **10** (9), 460–462.—By the use of a two-step gas-chromatographic method the separation and determination of the components of a mixture of acetaldehyde, acetylene, vinylacetylene, divinylacetylene, chloroprene, vinyl chloride, methyl vinyl ketone and dichlorobutene were possible. The columns were filled with granulated kieselguhr (0·3 to 0·4 mm) washed with H_2SO_4 and impregnated with 20% of di-(2-methoxyethyl) phthalate. Hydrogen was used as the carrier gas. J. ZÝKA

See also Abstracts—**898**, Cations in lubricating oils. **906**, Spectroscopically pure cyclohexane. **977**, Ge in coal. **1036**, Fe in paint driers. **1184**, Detection of functional groups in steroids. **1188**, Detection of org. compounds. **1217**, Determination of picrate, styphnate and picrolonate. **1236**, Hydroxybenzoic acids in mixtures. **1262**, N in materials containing nitrate. **1280**, Apparatus for elements in org. compounds. **1304**, Gas chromatography of volatile oils. **1308**, Modification of org. compounds before gas chromatography.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

1130. **New method of oxygen determination [in biological fluids].** A. Bracken, J. H. Glover and I. S. Longmuir (Brit. Oxygen Res. & Develop. Ltd., Deer Park Road, London, England). *Lancet*, 1960, **ii**, 411–412.—The method is based on a new principle of "potential interruption" in which the amount of charge lost by a platinum micro-electrode gives a measure of the O tension of the fluid in which it is immersed. The electrode is immersed permanently in saline soln. and is separated from the test soln. by a membrane of Teflon through which O diffuses rapidly to produce the same tension value on either side. The apparatus is sensitive to 0·02% of O and responds within 14 sec. H. F. W. KIRKPATRICK

1131. **Accurate and rapid complexometric serum potassium estimation.** W. Herbinger and W. Hubmayer (Allg. öffentlich. Krankenhaus., Linz, Austria). *Klin. Wochschr.*, 1960, **38** (16), 822–824.—To 1 ml of serum, free from haemolysis, add 2 ml of cobaltinitrite reagent and allow to stand at room temp. for 5 min. Centrifuge for 5 min. at 3000 r.p.m., decant the supernatant liquid, wash the ppt. with 5 ml of water, and re-centrifuge. Wash the ppt. twice more in a similar manner, then dissolve it in 3 drops of conc. H_2SO_4, dilute with 3 ml of water and quantitatively transfer the soln. to a 50-ml tube with 15 ml of water. Add 7 ml of 0·001M-EDTA (disodium salt), 12 drops of aq. NH_3 (sp. gr. 0·910) and a small amount of Eriochrome black T powder (1 in 100 parts of dry NaCl), and titrate the pale-blue soln. with 0·001M-$ZnSO_4$ to the violet end-point; serum potassium (milli-equiv. per litre) = (ml of EDTA − ml of $ZnSO_4$) × 2. E. C. BUTTERWORTH

1132. **Direct complexometric determination of calcium in urine.** J. J. M. H. Rutten and A. Groen (Lab. R. K. Ziekenhuis, Groningen, Netherlands). *Pharm. Weekbl.*, 1960, **95** (14), 449–452.—*Procedure*—Transfer 6 ml of a mixture of 50 ml of 0·5M-NaOH and 10 ml of 0·05M-trisodium citrate soln. to a Nessler tube. Add 0·2 ml of acidified urine and 2 drops of 0·1% aq. murexide soln. Titrate immediately with 0·008M-EDTA (disodium salt), using a colorimeter with a 585-mμ filter, until the extinction does not further increase. M. J. MAURICE

1133. **Rapid complexometric method for the estimation of calcium in bone, dentine and enamel.** J. A. Weatherell (Sch. of Dentistry, Univ. of Leeds, England). *Clin. Chim. Acta*, 1960, **5** (4), 610–612.—The method of Mori (*Anal. Abstr.*, 1960, **7**, 1471) is modified by addition of citrate buffer (pH 7) before titration to prevent pptn. of Ca at the high pH necessitated by the indicator (calcein). CDTA (1,2-diaminocyclohexane-*NNN′N′*-tetra-acetic acid) is used as the titrant. H. F. W. KIRKPATRICK

1134. **Determination of serum copper.** S. G. Welshman (Belfast City Hosp., N. Ireland). *Clin. Chim. Acta*, 1960, **5** (4), 497–498.—The method of Stark and Dawson (*Anal. Chem.*, 1958, **30**, 191) is adapted for use with serum. *Procedure*—To serum (1 ml) in a centrifuge tube add 2N-HCl (0·7 ml),

allow to stand for 15 min. then add 20% trichloroacetic acid (1 ml) and centrifuge at 2500 r.p.m. for 10 min. To the supernatant liquid (2 ml) add reagent mixture (conc. aq. NH_3 - satd. oxalyldihydrazide soln. - 40% acetaldehyde soln. - H_2O) (3:2:5:5) (3 ml), mix, and allow to stand for 15 min., then measure the colour at 542 mμ. A blank and standards (100 to 500 μg of Cu per 100 ml) are prepared as for the serum. The normal range found by this method was 85 to 163 μg of Cu per 100 ml of serum. H. F. W. Kirkpatrick

1135. **Detection of calcium-45 in bone solutions.** D. E. Pickering, H. L. Reed and R. L. Morris (Dept. of Biochem. and Pediatrics, Univ. Oregon Med. Sch., Portland, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1214–1215.—A method is described for counting ^{45}Ca in inorganic soln. of bone. The soln. are injected into a spiral plastic capillary phosphor assembly, from which aliquots may be recovered for the analysis of chemical components after the determination of the ^{45}Ca. Compared with solid counting systems this method has increased efficiency, eliminates self-absorption phenomena and reduces the sample-preparation time. K. A. Proctor

1136. **Flame-photometric determination of calcium in plants.** J. C. Brogan (Soil Lab., Johnstown Castle, Wexford, Eire). *J. Sci. Food Agric.*, 1960, **11** (8), 446–449.—Of the interfering substances found in plant material, PO_4^{3-}, SO_4^{2-} and Al^{3+} depress the emission of Ca at 622 mμ in a flame photometer with an air - propane - butane flame. The depression reaches a maximum, and then remains constant at ratios of Ca to interfering ion of 3:2 for P, 1:1 for S, and 1:1 for Al, and it is suggested that a refractory substance is formed. This is confirmed by the fact that lower maximum depressions are obtained at the same ratios with hotter flames. It is shown that an excess of SO_4^{2-} or PO_4^{3-} produces a maximum depression independent of small variations of either. The SO_4^{2-} and PO_4^{3-} have no effect on the depression caused by variable amounts of Al^{3+}. Since Al^{3+} occur in negligible quantity in plants other than tobacco, a procedure eliminating PO_4^{3-} and SO_4^{2-} interference by means of an excess of SO_4^{2-} was developed, in which dried plant material (0·5 g) is heated for 30 min. with 5 ml of conc. HNO_3 - conc. H_2SO_4 - 60% aq. $HClO_4$ (10:6:3), cooled and diluted to 50 ml. The emission is measured at 622 mμ and compared with standards similarly treated.

E. J. H. Birch

1137. **Direct-reading spectrometric determination of zinc, copper and lead in plant material.** A. Strasheim and D. J. Eve (Nat. Phys. Res. Lab., C.S.I.R., Pretoria, S. Africa). *Appl. Spectroscopy*, 1960, **14** (4), 97–100.—Samples are wet-ashed and the Zn, Cu and Pb are extracted in a $CHCl_3$ soln. of dithizone, with Cd added as an internal standard. The soln. is sprayed on to the lower edge of a disc, pressed from a 4:1 mixture of graphite and sodium carbonate, which rotates at 6 r.p.m. and carries the sample round into a 3·5-amp. interrupted d.c. arc struck between the upper edge and a graphite rod as the cathode. The relevant spectral lines are recorded by means of a direct-reading attachment to a Hilger medium spectrograph. Replicate analyses on a single sample gave coeff. of variation of 5·1% for Zn, 7·3% for Cu and 6·1% for Pb, with limits of detection of < 1 p.p.m., < 0·2 p.p.m. and < 0·1 p.p.m., respectively. Results were in reasonable agreement with those obtained on samples that had been analysed by other methods.

P. T. Beale

1138. **Tritium-labelled compounds. V. Radioassay of both carbon-14 and tritium in films with a proportional counter.** H. S. Isbell, H. L. Frush and N. B. Holt (Nat. Bur. Stand., Washington, D.C.). *J. Res. Nat. Bur. Stand., A*, 1960, **64** (4), 363–367.—The method is suitable for the measurement of as little as 0·001 μC of ^{14}C and 0·005 μC of ^{3}H in water-soluble non-volatile compounds. The sources are prepared by evaporating the soln. over an area of 5 sq. cm on a stainless-steel planchette. Special features of the technique include the addition of *O*-(carboxymethyl)cellulose to the soln. to prevent crystallisation, special cleaning of the planchette to ensure ready spreading of the soln., and the conditioning of the film in the atmosphere over a satd. soln. of K acetate. The sources are counted in a 2π gas-flow counter, and the activities of ^{14}C and ^{3}H are derived from two measurements, one being taken with a screen of thickness 0·8 mg per sq. cm over the source; the screen completely absorbs all the radiation from ^{3}H, but allows the passage of ≃50% of that from ^{14}C. G. J. Hunter

1139. **Micro-determination of soluble silica in urine.** J. Paul (Dept. of Med. Biochem., Univ., Birmingham, England). *Biochem. J.*, 1960, **77** (1), 202–205.—The method, which is accurate and reproducible, can be used for the determination of trace amounts of SiO_2 in rat and human urine in the presence of high concn. of phosphate. Most of the phosphate present is first pptd. by $CaCl_2$ - aq. NH_3 at pH 7·0, and the filtrate is then treated with ammonium molybdate - aq. HCl; this converts the residual phosphate into molybdophosphate, which is selectively extracted with ethyl acetate. The molybdosilicic acid in the aq. phase is then reduced to molybdenum blue by 1-amino-2-naphthol-4-sulphonic acid in the presence of citric acid, and the extinction of the blue soln., which is linear wth respect to Si concn. in the range 5 to 60 μg of SiO_2, is measured at 690 mμ. J. N. Ashley

1140. **Mineralisation of biological material containing radioactive sulphur by a mixture of nitric and hydrochloric acids.** Z. Franc (Res. Inst. Pharm. and Biochem., Prague). *Chem. Listy*, 1960, **54** (9), 971–973.—*Procedure*—Mix the biological material (blood, urine, etc.) with 10 ml of oxidation mixture [conc. HNO_3 - conc. HCl (5:1)], add satd. $CuSO_4$ soln. as catalyst (1 drop) and set aside for 1 to 2 hr. Then heat on a sand bath, and after 3 to 4 hr. evaporate the clear soln. to dryness. Dissolve the residue in a few drops of conc. HCl and H_2O (3 ml). If the residue is black it must be treated again with oxidation mixture. To the clear blue - green soln. add benzidine reagent (dissolve 5 g of benzidine hydrochloride by heating in 40 ml of N-HCl and dilute with 500 ml of 70% ethanol) (5 ml) and set aside for 3 hr. Collect the ppt. on paper in a sintered glass filter and wash with ethanol (96%) (5 ml). Place the wet paper on a Novodur holder and after drying measure the activity with a Geiger - Müller counter. J. Zýka

1141. **Spectrophotometric determination of vanadium in biological material.** F. H. Hulcher (Bowman Gray School of Med., Wake Forest Coll., Winston-Salem, N.C., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1183–1185.—The procedure described is applicable to the routine determination of 0·2 to

50 μg of V in animal tissue after wet oxidation with HNO_3 - $HClO_4$ to ensure the presence of V^{5+}. Interference by Fe is avoided by the pre-extraction of its benzohydroxamic acid complex from aq. soln. into octyl alcohol at pH 8·5. Several extractions are made, a mixture of octyl alcohol and benzene being used if benzohydroxamic acid remains at the interface. The V in the aq. phase is then extracted into octyl alcohol at pH 3 (0·1M-glycine - HCl buffer) and the extinction of the organic phase is measured at 450 mμ in a 1-cm cell. Titanium (in feed, faeces or plant material) should be removed after extraction of the Fe. Cobalt interferes at concn. > 100-fold that of the V; a slight modification for samples of bone facilitates the separation of vanadate from Ca. The error is $\simeq$0·9% for 2 to 10 μg of V in 2 ml of solvent. W. J. Baker

1142. **Determination of tungsten in biological material by activation analysis.** H. J. M. Bowen (Wantage Radiation Lab., Grove, Berks., England). *Biochem. J.*, 1960, **77** (1), 79–82.—The method is very much more sensitive and more selective than other methods for the determination of W in biological material. There is no interference from Mo, and the theoretical sensitivity is 50 $\mu\mu$g of W; in practice 1 mμg of W can be determined with an accuracy of ±10%. After being irradiated by neutrons, the material is wet-ashed, and the ^{187}W is separated chemically from other radioactive elements produced by the activation. The ^{187}W is finally pptd. as the benzoin α-oxime derivative, which is ignited to give $^{187}WO_3$, and the β-ray emission is then measured with an end-window Geiger counter. The whole chemical procedure requires approx. 3 hr. for 8 samples. J. N. Ashley

1143. **X-ray spectroscopy in the chemical laboratory. II. Chlorine and sulphur; automatic analysis of ultra-micro samples.** S. Natelson and B. Sheid (Roosevelt Hosp., New York, N.Y., U.S.A.). *Clin. Chem.*, 1960, **6** (4), 299–313.—Serum samples (20 to 25 μl) are dried in confined spots on a long strip of filter-paper which is passed continuously through the X-ray field. Methods are described in detail.

III. Sulphur distribution in the electrophoretic protein fractions of human serum; abnormalities observed in certain disease states. S. Natelson and B. Sheid. *Ibid.*, 1960, **6** (4), 314–326.—A method of determining the sulphur to protein ratio in separated fractions is described. H. F. W. Kirkpatrick

1144. **Determination of manganese in biological material.** H. Holešovská-Kozáková (Inst. Hyg. and Ind. Diseases, Prague). *Chem. Listy*, 1960, **54** (9), 967–970.—The method, which is suitable for the determination of Mn in serum albumin without mineralisation, is based on the reaction of Mn with formaldoxime in an alkaline medium. Samples of urine must be mineralised. *Procedure*—To the sample of urine (5 to 10 ml) add an equal vol. of HNO_3, evaporate to dryness and repeat the treatment till the residue remains white (6 × 3 ml of HNO_3). If necessary, add 0·1 ml of conc. H_2SO_4 before mineralisation is complete. Dissolve the residue in a small vol. of H_2O and neutralise with aq. NH_3. Add $(NH_4)_2SO_4$ soln. (50%) (1 ml), formaldoxime soln. [dissolve paraformaldehyde (3 g) and hydroxyammonium chloride (7 g) in H O (15 ml) by gentle heating] (0·2 ml) and aq. NH_3 (25%) (1 ml) and dilute with H_2O to 10 ml. Measure the extinction at 450 mμ and compare with a calibration curve prepared for the range 1 to 3 μg of Mn per ml. J. Zýka

1145. **Note on suitable solvent systems usable in the liquid scintillation counting of animal tissues.** G. A. Bruno and J. E. Christian (Bionucleonics Dept., Purdue Univ., Lafayette, Ind., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (8), 560–561.—To prepare a colourless molar soln. of methylbenzethonium hydroxide, dissolve the purified chloride (48 g) in methanol (50 ml), add 8% methanolic NaOH soln. (50 ml) and filter. Add 2 ml of this soln. to the tissue sample (75 mg) and break up the tissue with a glass rod or, better, vibrate ultrasonically at 400 kc/s for 1 hr.; heat at 100° until dissolution is complete, cool, dilute with scintillator soln. and count the ^{14}C β-emission. Little or no colour develops during this procedure, and the absolute counting efficiency is as high as 68%. A. R. Rogers

1146. **Ultra-violet photometric assay of thiopentone and pentobarbitone in blood and plasma.** S. I. Oroszlan and G. D. Maengwyn-Davies (Sch. of Med., Georgetown Univ., Washington, D.C.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (8), 507–509.—Use of an Allen-type correction procedure (*J. Clin. Endocrin.*, 1950, **10**, 71) makes possible the quant. determination of thiopentone (**I**) or pentobarbitone (**II**) at concn. of $\simeq$20 μg per ml. Recovery results show an average of 99·1% with a standard error of 5·4% (6 samples), in comparison with 95% with a standard error of 10·5% (4 samples) by the method of Goldbaum (*Anal. Chem.*, 1952, **24**, 1604). *Procedure*—Mix the sample (4 ml) with 0·85% NaCl soln. (0·1 ml), shake mechanically with $CHCl_3$ (40 ml) for 30 min. and filter the $CHCl_3$ layer through Whatman No. 1 paper. Vigorously shake a 20-ml aliquot of the filtrate by hand for 1 min. with 0·45N-NaOH (for **I**) or 0·2N-NaOH (for **II**) (4 ml), allow to separate and measure the u.v. absorption of the upper layer. Calculate $E_{305} - 0{\cdot}5E_{295} - 0{\cdot}5E_{315}$ for **I**, or $E_{255} - 0{\cdot}5E_{245} - 0{\cdot}5E_{265}$ for **II**. A. R. Rogers

1147. **Urine colour test for the detection of phenothiazine compounds.** I. S. Forrest and F. M. Forrest (V. A. Hosp., Brockton, Mass., U.S.A.). *Clin. Chem.*, 1960, **6** (4), 362–363.—Several potentially interfering factors have been encountered in extending the test previously described (*Anal. Abstr.*, 1960, **7**, 3874) to patients other than neuropsychiatric. Patients with phenylketonuria with excretion levels of 15 to 90 mg per day consistently showed false positive colours, as also did patients on 6 g or more per day of 4-aminosalicylic acid. Low-level positive tests should be interpreted with care. No false negative results have been obtained. H. F. W. Kirkpatrick

1148. **Chemical determination of laevomycetin in biological liquids.** V. P. Sil'vestrov and A. L. Yaroslavtsev (Kirov Military Medical Acad., USSR). *Lab. Delo*, 1960, **6** (4), 20–23.—*Procedure*—Add 3% $Ba(OH)_2$ soln. (2 ml) and 5% $ZnSO_4$ soln. (2 ml) to blood or bile (1 ml), shake the tube, set it aside for 10 min. at room temp., and then centrifuge the contents at 3000 r.p.m. for 10 min. Add 70% HCl (0·5 ml) and some granulated zinc to the clear supernatant liquid (3 ml), place the tube in a boiling-water bath for 30 min., then allow it to cool in a cold-water bath. Remove the zinc, add 0·15% $NaNO_2$ soln. (0·5 ml), shake the tube, set it aside

for 5 min., add 0·5% ammonium sulphamate soln. (2 ml), and after 3 min. add 0·2% *N*-1-naphthylethylenediamine dihydrochloride soln. (0·5 ml); set the soln. aside for 40 min. and measure the colour photometrically against a control at 530 mμ. Calculate the antibiotic concn. from a standard curve of extinction *vs.* laevomycetin concn. (3 to 30 μg); linearity is maintained in this range. The method cannot be applied to urine.

K. R. Cook

1149. **Toxicological studies on hydrocarbons. VI. Colorimetric method for the determination of kerosine in blood.** H. W. Gerarde and P. Skiba (Med. Res. Div., Esso Res. and Engng Co., Lindon, N.J., U.S.A.). *Clin. Chem.*, 1960, **6** (4), 327–331.—Add 5 ml of blood directly from a syringe to 35 ml of 0·1N-HCl in a 2-oz wide-mouthed bottle, cap the bottle and mix by inversion. Add 5 ml of CCl_4 and shake vigorously for 3 to 5 min. Take off the aq. layer, centrifuge the CCl_4 soln. and remove any residual aq. layer. Transfer the CCl_4 soln. to a stoppered test-tube, add 5 ml of formaldehyde - H_2SO_4 reagent (1 ml of 40% formaldehyde soln. mixed with 100 ml of conc. H_2SO_4), stopper the tube and shake vigorously for 2 min. Centrifuge the tube for 5 min. at 2000 r.p.m. and measure the colour at 490 mμ. A standard curve is prepared with CCl_4 soln. containing 10 to 100 p.p.m. of kerosine.

H. F. W. Kirkpatrick

1150. **Determination of sugars and related substances on glass-fibre paper.** G. Jayme and G. Hahn (Inst. f. Cellulosechem. mit Holzforsch., Tech. Hochschule, Darmstadt, Germany). *Angew. Chem.*, 1960, **72** (15), 520–522.—Separate the reducing sugars, uronic acids and similar substances by chromatography on glass-fibre paper (*cf.* Jayme and Knolle, *Anal. Abstr.*, 1956, **3**, 2773), and analyse the cut-out zones by oxidation with excess of hot acid $K_2Cr_2O_7$ soln. followed by iodimetric back-titration. Perform a blank determination on sugar-free glass-fibre paper and a calibration experiment with a known amount of glucose. The accuracy is better than ±5%.

A. R. Rogers

1151. **Triple-test strip for urinary glucose, protein and pH.** H. M. Free, G. F. Collins and A. H. Free (Ames Res. Lab., Elkhart, Ind., U.S.A.). *Clin. Chem.*, 1960, **6** (4), 353–361.—The reactive end of a strip of special stiff cellulose is separated into three areas by water-impervious barriers. Protein is tested for by the protein error of indicators, glucose by glucose oxidase, and pH by a mixture of indicators. The three tests are made simultaneously by dipping the strip in the urine.

H. F. W. Kirkpatrick

1152. **Enzymic fluorimetric methods for the microdetermination of hexose phosphates in muscle.** K. Seraydarian, W. F. H. M. Mommaerts and A. Wallner (Dept. of Med., Los Angeles County Heart Assoc. Cardiovascular Res. Lab., Univ. of Calif., U.S.A.). *J. Biol. Chem.*, 1960, **235** (8), 2191–2195.—The Slater methods (*Biochem. J.*, 1953, **53**, 157) for the enzymic determination of hexose phosphates and triose phosphates are adapted to fluorimetric measurement. The recoveries of these glycolysis intermediates and the accuracy of the method are within a few per cent. of the theoretical values. The determination requires the use of only a small amount of an extract of 100 mg of muscle.

J. N. Ashley

1153. **The determination of amyloid in plant seeds.** P. Kooiman (Tech. Univ., Delft, Netherlands). *Rec. Trav. Chim. Pays-Bas*, 1960, **79** (7), 675–678.—Amyloids give a blue colour in very dilute soln. with KI_3 provided that an excess of K_2SO_4 is present. Halides interfere. *Procedure*—Grind 1 g of seed containing ≃50% of amyloid and add 150 ml of water and 10 ml of 2N-NaOH, leave for 24 hr. at 25° with occasional stirring, centrifuge and repeat the extraction twice. Combine the extracts, neutralise with dil. H_2SO_4, filter through kieselguhr, wash the residue and make the filtrate up to 1 litre. To 1 ml of the soln. (containing 0·05 to 0·3 mg of amyloid) add 0·5 ml of iodine soln. (0·5 g of iodine and 1 g of KI in 100 ml of H_2O) and 5 ml of 20% K_2SO_4 soln. Mix by shaking and read the extinction after 60 min. at 640 mμ against a reagent blank.

B. Heining

1154. **Determination of small quantities of 3-furaldehyde in blood and urine.** G. N. Nazyrov and Kh. Ya. Vengerskaya (Uzbek Scientific-Study Sanitary Inst.). *Lab. Delo*, 1960, **6** (5), 35.—*Procedure*—Thoroughly mix 0·5 ml of blood with 5 ml of a 3·5% soln. of metaphosphoric acid, allow to stand for 40 to 50 min. and filter. Make up the vol. to 7 ml with water, and determine the 3-furaldehyde colorimetrically with aniline acetate. Strongly coloured urine is first decolorised with a 40% soln. of Pb acetate.

A. Burwood-Smith

1155. **New quantitative modification of the glyoxylic acid reaction.** H. P. Rieder and M. Böhmer (Psychiat. Clinic, Univ., Basel, Switzerland). *Clin. Chim. Acta*, 1960, **5** (4), 520–524 (in English).—Contrary to the findings of Velek and Valentova (*J. Nerv. Ment. Dis.*, 1959, **128**, 184), glyoxylic acid produces a specific reaction with indoles in the presence of H_2SO_4. This reaction is enhanced by a low concn. of $FeCl_3$. The quant. method based on this reaction has been previously described by the authors (*cf. Helv. Chim. Acta*, 1959, **42**, 1793).

H. F. W. Kirkpatrick

1156. **Fluorimetric determination of gentisic acid in blood and urine.** M. Nepraš (Centr. Lab. Kúnz, Pardubice, Czechoslovakia). *Českosl. Farm.*, 1960, **9** (7), 346–347.—*Procedure*—To 2 ml of blood serum or urine in a centrifuge-tube, diluted 1:100 with H_2O, add 7N-H_2SO_4 (2 ml) and ethyl ether (10 ml). Extract and evaporate 5 ml of the ether extract on a water bath. Dilute the residue with H_2O (10 ml) and measure the fluorescence in a Pulfrich photometer (filter L_{420}). Compare with a calibration curve. The method is suitable for samples containing ≯80 μg of gentisic acid per 1 ml.

J. Zýka

1157. **Determination of citrulline in plasma.** M. P. Mockler, L. R. Menebroker, F. L. Humoller and A. J. Barak (V.A. Hosp., Omaha, Nebr., U.S.A.). *Clin. Chem.*, 1960, **6** (4), 291–298.—Serum is incubated with urease to destroy urea, and the proteins are pptd. with picric acid and separated by centrifuging. The filtrate is passed through a column of Amberlite CG-400, Type 1, to remove excess of picric acid, and the percolate drips directly on to another column containing Amberlite CG-120, Type 2, 200 mesh, on which the citrulline is adsorbed. The citrulline is eluted with 15% NaCl soln. The colour test is carried out on the eluate with a H_2SO_4 - H_3PO_4 mixture, diacetyl monoxime soln. and phenylhydrazine soln. (*Cf.* Fearon,

Biochem. J., 1939, **33**, 902.) A normal range of 4·3 to 9·2 μg per 100 ml was obtained.
H. F. W. KIRKPATRICK

1158. **Rapid demonstration of haemoglobin A_2 by means of agar-gel electrophoresis.** V. J. Yakulis, P. Heller, A. M. Josephson and L. Singer (Veterans Admin. West Side Hosp., Chicago, Ill.). *Amer. J. Clin. Path.*, 1960, **34** (1), 28–34.—Haemoglobin soln. obtained from the blood of normal subjects and from the blood of patients with thalassaemia are submitted to electrophoresis on agar gel for 90 min. at 90 to 100 V and 40 to 50 mA. After drying, the electropherograms are stained for 30 min. with Amido black 10B (C.I. Acid Black 1) soln. and measured in a densitometer at 500 mμ. Good separation of haemoglobins A_1 and A_2 was achieved and up to 3 unidentified non-haemoglobin fractions were observed. Haemolysates from 80 normal subjects gave a mean haemoglobin A_2 content of 6·7 ± 1·4%, and from 19 thalassaemics a mean of 14·7 ± 2·6%.
D. E. EVANS

1159. **Note on the haemoglobin error in some non-precipitation diazo methods for bilirubin determinations.** D. Watson (Royal Women's Hosp., Melbourne, Australia). *Clin. Chim. Acta*, 1960, **5** (4), 613–615.—The method of Lathe and Ruthven (*Anal. Abstr.*, 1958, **5**, **3834**) gives no significant error in bilirubin assay in the presence of 1% of haemoglobin, and is recommended for the analysis of moderately haemolysed specimens. Other methods investigated are subject to considerable error in the presence of haemoglobin.
H. F. W. KIRKPATRICK

1160. **Spectrophotometric method for the determination of methaemoglobin in blood.** G. E. Martin, J. I. Munn and L. Biskup (Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 743–746.—Methaemoglobin has a characteristic absorption peak at 630 mμ. Buffered haemoglobin soln. from whole blood is measured for extinction at 630 mμ before and after addition of sodium cyanide. Another fraction of buffered haemolysed red-cell soln. is centrifuged, and the supernatant liquid treated with $K_3Fe(CN)_6$. Finally the total haemoglobin content is determined (Crosby *et al.*, *U.S. Armed Forces Med. J.*, 1954, **5**, 693). The concn. of methaemoglobin can then be calculated.
A. A. ELDRIDGE

1161. **Determination of chromoproteins by the radiometric method.** A. A. Shatalova, G. I. Meerov and Ya. R. Savinskiĭ (Psychoneurology Res. Inst., Leningrad). *Biokhimiya*, 1960, **25** (4), 577–583.—*Procedure*—Prepare a stabilising mixture from Na oxalate (400 mg), $Na_2S_2O_4$ (1000 mg) and saponin (400 mg). Add stabilising soln. (18 mg of mixture per ml of H_2O) (0·2 ml) to blood (0·2 ml) and saturate the blood with radioactive carbon monoxide (sp. activity ≮1·4 μC per ml) by bubbling it very slowly with ^{14}CO - air for 2 min. Determine the mean activity of the blood (0·1 ml) (3 determinations) during 3 min. by means of a radiation counter. The results for haemoglobin are in good agreement with those obtained by the photometric method; the relative error is ≯ ±3%. It is possible to determine haemoglobin with an accuracy within ±6%.
K. R. COOK

1162. **Chromatographic - spectrophotometric method for the determination of caeruloplasmin.** H. Deutsch (Dept. of Physiol. Chem., Univ. of Wisconsin, Madison, U.S.A.). *Clin. Chim. Acta*, 1960, **5** (4), 460–463.—This protein fraction is separated from other serum constituents on diethylaminoethyl cellulose (Petersen and Sober, *J. Amer. Chem. Soc.*, 1956, **78**, 751). After elution, the blue soln. is measured before and after reduction with ascorbic acid.
H. F. W. KIRKPATRICK

1163. **Spectrophotometric determination of chlorophylls and pheophytins in plant extracts.** L. P. Vernon (Brigham Young Univ., Provo, Utah, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1144–1150.—Samples are dissolved in 80% acetone and absorptivities are observed for chlorophyll *a*, chlorophyll *b*, pheophytin *a* and pheophytin *b* at appropriate wavelengths. Equations are derived from the changes in absorptivities, from which the individual concentrations are calculated.
G. P. COOK

1164. **Determination of choline and betaine in the presence of each other.** O. Hrdý and S. Lochmanová (State Inst. for Control of Drugs, Prague). *Českosl. Farm.*, 1960, **9** (7), 335–339.—The anionic component of choline chloride or tartrate and of betaine hydrochloride is separated on Amberlite IRA-400, and the eluted bases are then separated by means of Amberlite IRC-50. The betaine (eluted with H_2O) is determined by evaporating the eluate to dryness and titrating in anhyd. acetic acid with 0·05N-$HClO_4$ to methyl violet. The choline is eluted from the column with N-HCl, and pptd. and weighed as the reineckate. For mixtures of choline with betaine in the ratio 1:10, and vice versa, an accuracy of ±4% was achieved.
J. ZÝKA

1165. **Polarographic determination of adrenochrome semicarbazone.** M. Sterescu and M. Popa. *Rev. Chim., Bucharest*, 1960, **11** (8), 489.—The sample soln. containing 0·02 g of adrenochrome semicarbazone is treated with 1 ml of 1% gelatin soln. and diluted to 100 ml with 10% LiCl soln. Oxygen is removed by a stream of N and the polarogram is measured between 0·0 and −1·0 V. Errors are ≯ ±1%.
H. SHER

1166. **Fully automatic quantitative determination of amino-acids.** G. Braunitzer (Max-Planck-Inst. f. Biochem., München, Germany). *Angew. Chem.*, 1960, **72** (14), 485–489.—The experiments of Moore *et al.* to separate amino-acid mixtures by ion exchangers led to the fully automatic apparatus developed by Spackmann *et al.* (*Anal. Abstr.*, 1958, **5**, 4359). To speed up the exchange in the columns, the size of the granules is decreased and the temp. increased. The eluate is mixed continuously with ninhydrin soln. in constant-volume proportion, the mixture is passed through a Teflon coil, the length of which is calculated so that the eluate plus ninhydrin remain in the water bath for exactly 15 min., during which the reaction takes place. The mixture then passes a photocell which measures the extinction continuously, the values being registered at 4-sec. intervals on log paper.
I. DICKINSON

1167. **Infra-red spectra of amino-acids in water.** F. S. Parker and D. M. Kirschenbaum (Dept. of Biochem., State Univ. of New York, U.S.A.). *Spectrochim. Acta*, 1960, **16**, 910–917.—The range 6·5 to 11·0 μ is available for soln. of amino-acids in water, and assignments are offered for bands occurring in the spectra of the 36 common amino-acids examined. It is possible to distinguish

between most of the individual members of a group in the case of the 13 aliphatic amino-acids examined, the sulphur-containing acids and the basic amino-acids. Sarcosine, (±)-pipecolic acid, L-proline and L-hydroxyproline are also identifiable. The method is not successful for such compounds as DL-valine and the aromatic amino-acids, which are relatively insoluble, or for the acidic amino-acids examined, which all give similar spectra. E. G. CUMMINS

1168. **Polarographic determination of dinitrophenylamino-acids.** I. A. Vaĭntraub (State Univ., Kishinev, USSR). *Biokhimiya,* 1960, **25** (4), 688–692.—Paper chromatograms of dinitrophenylamino-acids were sectioned and the sections placed in a Britton - Robinson or borate buffer soln. (1 ml) at pH 8·2 to 8·3 for 20 min. To remove O, Na_2SO_3 (10 to 20 mg) was added to each soln., an aliquot (0·5 to 1·0 ml) of which was measured polarographically after 3 to 4 min. An electronic integrating - differentiating polarograph was used; the polarographic cell with an external anode (S.C.E. half-cell) (Sochevanov, *Zavod Lab.*, 1954, **20**, 992) was thermostatically controlled at 25° ± 0·1°. The drop time of the capillary was determined at a potential of −0·8 V. Di(dinitrophenyl)histidine, in contrast to all the other dinitrophenylamino-acids, which undergo a two-step reduction, formed three waves when polarographed under these conditions. This permits its determination even in the presence of an 80-fold excess of all the other amino-acids. K. R. COOK

1169. **New method for the quantitative determination of urinary β-aminoisobutyric acid (3-amino-2-methylpropionic acid).** G. B. Gerber and G. Gerber (Div. of Exp. Radiol., Sch. of Med., Univ. Rochester, N.Y., U.S.A.). *Clin. Chim. Acta,* 1960, **5** (4), 607–608.—The compound is converted into the dinitrophenyl derivative, which is chromatographed on paper with heptane - pyridine (7:3) as solvent system, located with u.v. light, eluted with 0·3% aq. $NaHCO_3$ soln. and measured at 360 mμ. H. F. W. KIRKPATRICK

1170. **Simplified method for detecting formimidoylglutamic acid in urine as a test of folic acid deficiency.** J. P. Knowles, T. A. J. Prankerd and R. G. Westall (Univ. Coll. Hosp. Med. Sch., London, England). *Lancet,* 1960, **ii**, 347–348.—Excretion of formimidoylglutamic acid (**I**) after histidine loading is detected by high-voltage electrophoresis on Whatman 3MM paper at 6·0 kV and 60 to 80 milliamp. for 20 min. with a buffer soln. of pyridine (50 ml), glacial acetic acid (20 ml) and H_2O to 5 litres (pH 5·4). The spots can be detected by spraying with nitroprusside - ferricyanide reagent (Kirby-Berry *et al., Texas Univ. Publ.*, 1951, No. 5509, p. 22) to give an immediate orange - red stain; alternatively, duplicate papers are run and one is suspended in a closed beaker containing conc. aq. NH_3, which converts **I** into glutamic acid. The paper is dried at 110° for 10 min., dipped in acetone - acetic acid (23:2) and re-dried at 110° for 30 sec. Both papers are stained with ninhydrin, and 24 hr. later are fixed with 0·25M-$NiSO_4$. **I** shows a spot on the strip that has been treated with aq. NH_3, but not on the other. Patients are given 20 g of histidine orally and the urine is collected in fractions, the 5 to 6-hr. fraction showing maximum excretion of **I**. A dilution of the urine 1 to 10 with H_2O showing **I** when treated as described above is indicative of folic acid deficiency.
H. F. W. KIRKPATRICK

1171. **Stability of colour developed by the Sakaguchi reaction of arginine.** K. R. Bhattacharya (Indian Inst. for Biochem. and Exp. Medicine, Calcutta 13). *Ann. Biochem., Calcutta,* 1960, **20** (3), 57–64.—Factors affecting colour stability when using urea for destroying excess of hypohalite have been examined with particular reference to pH, temp. and reaction time. The presence of alkali hydroxide inhibits the destruction of traces of hypohalite which results in colour fading. This does not occur on filter-paper owing to atmospheric carbonation of the alkali. Reduction of the pH with bicarbonate or the use of 2% $Na_2S_2O_5$ or $Na_2S_2O_3$ soln. in place of urea soln. also increases the colour stability. P. M. KINGSTON

1172. **Estimation of thyroxine in human plasma by an electrophoretic technique.** R. P. Ekins (Middx. Hosp. Med. Sch., London, England). *Clin. Chim. Acta,* 1960, **5** (4), 453–459.—The method described depends on measurement of the distribution of thyroxine between albumin and thyroxine-binding protein after addition of exogenous ^{131}I-labelled thyroxine *in vitro*.
H. F. W. KIRKPATRICK

1173. **Determination of urine protein.** H. W. Marlow (V.A. Hosp., Downey, Ill., U.S.A.). *Clin. Chem.*, 1960, **6** (4), 341–344.—The following method is recommended. An aliquot of a 24-hr. sample of urine is diluted 1 to 10, and 0·2 ml is mixed with 3·8 ml of H_2O and 1 ml of biuret reagent. After 15 min., 1 ml of Folin - Ciocalteau reagent, diluted with an equal vol. of H_2O, is added and after another 15 min. exactly the colour is read at 700 mμ against a blank run concurrently with 0·2 ml of urine and 4·8 ml of H_2O. The extinction × 1490 gives protein concn. in mg per 100 ml if a 19-mm cell is used. The biuret reagent is prepared by mixing 45 ml of a soln. containing 10 g of NaOH, 40 g of Na_2CO_3 and 1 g of Na K tartrate per litre with 5 ml of 0·5% $CuSO_4.5H_2O$ soln.
H. F. W. KIRKPATRICK

1174. **Quantitative staining of protein fractions in paper electrophoresis.** J. Masopustand and J. Homolka (Forschungsinst. f. Kindesentwicklung, Charles' Univ., Prague, Czechoslovakia). *Clin. Chim. Acta,* 1960, **5** (4), 464–470 (in German).—Factors influencing the staining of proteins with bromophenol blue are investigated. The recommended method is as follows. Dry the Whatman No. 1 paper strips at 110° for 30 min. and immerse in a bath of dye (bromophenol blue 2 g, glacial acetic acid 100 ml, and 96% ethanol to 1 litre) for 20 min. Wash 3 times by agitation for 2 min. in 2% acetic acid, and dry. Elute the separate fractions with 0·01N-NaOH and read the colour at 590 mμ. H. F. W. KIRKPATRICK

1175. **Improved technique for the quantitation of serum haptoglobin.** J. Javid and H. Horowitz (Dept. of Haematol., Montefiore Hosp., New York). *Amer. J. Clin. Path.*, 1960, **34** (1), 35–39.—A mixture of the test serum with a standard haemoglobin soln. was submitted to electrophoresis on paper with phosphate buffer (0·05M, pH 7·0) for 14 hr. at 135 V. After drying, the electropherograms were stained with 3,3′-dimethoxybenzidine for 10 min. and scanned in a densitometer. The haptoglobin content of each serum was also determined by Nyman's method and a correlation of 0·98 was obtained over a range from 0 to 500 mg per 100 ml. (*Cf. Scand. J. Clin. Lab. Invest.*, 1959, **11**, Suppl. 39, 1.) D. E. EVANS

1176. **Chromatographic separation of serum lipoproteins on glass-powder columns.** L. A. Carlson (Karolinska Hosp., Stockholm, Sweden). *Clin. Chim. Acta*, 1960, **5** (4), 528–538 (in English).—Ordinary soda glass, 100 to 140 mesh, is used. The lipoproteins are adsorbed at pH 8·8 and eluted in fractions by stepwise increase in pH. The fractions are extracted and analysed by conventional methods. Results are presented and discussed.

H. F. W. Kirkpatrick

1177. **Carbohydrates in protein. II. Hexose, hexosamine, acetyl and amide-nitrogen content of hen's-egg albumin. [Determination of hexosamine.]** P. G. Johansen, R. D. Marshall and A. Neuberger (Dept. of Chem. Path., St. Mary's Hosp. Med. Sch., London, England). *Biochem. J.*, 1960, **77** (2), 239–247.—A modification of the Cessi method (*Brit. Abstr. C*, 1953, 126) is used for the determination of glucosamine. It depends on the formation and steam-distillation of a volatile chromogen (2-methylpyrrole) which condenses with Ehrlich's reagent to give a product with max. absorption at 550 mμ. The method is more specific than the method of Elson and Morgan (*Biochem. J.*, 1933, **27**, 1824), and decreases the interference that is observed when neutral sugars and amino-acids are heated together. The new method is applicable only to 2-substituted, and not 3-substituted, glucosamines.

J. N. Ashley

1178. **Rapid determination of cholesterol bound to the α- and β-lipoproteins of serum.** M. Burstein and J. Samaille (Nat. Transfusion Centre, Longuine, Paris, France). *Clin. Chim. Acta*, 1960, **5** (4), 609 (in French).—β-Lipoproteins are pptd. by addition of heparin and manganese chloride. The α-lipoproteins are separated by electrophoresis of the supernatant fluid after centrifuging, and the cholesterol is determined. The total cholesterol in the serum is determined and the difference gives the cholesterol bound to the β-fraction.

H. F. W. Kirkpatrick

1179. **Determination of oestrogens in urine by the method of Ittrich.** H. Breder and J. Gertz (Univ.-klinik u. Poliklinik, Bonn, Germany). *Clin. Chim. Acta*, 1960, **5** (4), 544–551 (in German).—The method of Ittrich (*Hoppe-Seyl. Z.*, 1958, **312**, 1) is satisfactory for pregnancy urines, but the fluorimetric method is preferred for urines low in oestrogen.

H. F. W. Kirkpatrick

1180. **Method for the quantitative fractionation of mixtures of 2-methoxyoestrone, oestrone, ring D α-ketolic oestrogens, 17β-oestradiol, 16-epioestriol and oestriol by partition chromatography and the Girard reaction.** M. L. Givner, W. S. Bauld and K. Vagi (Dept. of Metabol., Montreal Gen. Hosp., Canada). *Biochem. J.*, 1960, **77** (2), 400–406.—A method for the separation and determination of pure 2-methoxyoestrone, oestrone, 16α-hydroxyoestrone, 17β-oestradiol, 16-epioestriol and oestriol is described. The steroids are separated into ketonic and non-ketonic fractions by means of Girard P reagent, the two fractions are chromatographed on Celite and each steroid is eluted with appropriate solvent mixtures. The steroids are then determined colorimetrically by the Kober reaction.

J. N. Ashley

1181. **Chemical method for the quantitative determination of 2-methoxyoestrone, oestrone, ring D α-ketolic oestrogens, 17β-oestradiol, 16-epioestriol and oestriol in human urine.** M. L. Givner, W. S. Bauld and K. Vagi (Dept. of Metabol., Montreal Gen. Hosp., Canada). *Biochem. J.*, 1960, **77** (2), 406–415.—The method involves enzymic hydrolysis of the urine, with gluculase from *Helix pomatia* for pregnancy urine, and bacterial β-glucuronidase for male urine, followed by (*i*) extraction of the steroids with ethyl ether, separation of the ketonic from the non-ketonic oestrogens by reaction with Girard P reagent, (*ii*) purification of the non-ketonic fraction by heating under reflux with alkali, (*iii*) separation and purification of the 17β-oestradiol, 16-epioestriol and oestriol fractions by column chromatography on Celite, (*iv*) purification of the 17β-oestradiol fraction by heating under reflux with alkali, (*v*) separation and purification of the 2-methoxyoestrone, oestrone and ring D α-ketolic oestrogen fractions by column chromatography on Celite, (*vi*) purification of the 2-methoxyoestrone fraction by heating under reflux with alkali and (*vii*) purification of the oestrone fraction by washing with alkali. The purified fractions are determined spectrophotometrically by the Kober reaction, with correction for non-oestrogen chromogenic substances. The six fractions can be determined in as little as 20 ml of late-pregnancy urine, and the ring D α-ketolic oestrogens and oestriol can be measured in 100 ml of male urine. Greater sensitivity could be achieved by the use of fluorimetric methods. In eight working days one person can determine the oestrogen content of four urines in duplicate.

J. N. Ashley

1182. **Paper chromatography of urinary corticosteroids.** C. Matarazzo, M. L. Orabona, R. Chiummo and G. Sacchetti (Ist. di Patologia Speciale Medica, Univ. Bari, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1960, **36** (16), 863–866.—Urinary corticosteroids are separated by paper chromatography and determined colorimetrically from extracts prepared and purified according to the method of Orabona and Matarazzo (*Ibid.*, 1958, **34**, 1236). The sample extract in $CHCl_3$ (2 $\times$ 0·1 ml) is applied as a band to a strip (40 cm $\times$ 2·5 cm) of Whatman No. 1 paper previously impregnated with a 20% soln. of propane-1,2-diol in acetone. A soln. containing cortisone (50 μg) and hydrocortisone (50 μg) (as free alcohols) is applied to a similar strip and the chromatograms are developed for 72 hr. by the descending technique with a mixture of toluene and propane-1,2-diol. The percolates are collected from the bottom of the papers, evaporated to dryness in a current of air and re-chromatographed for 20 hr. by the same technique and with an additional parallel standard of deoxycortone (50 μg in ethanol). The developed strips are cut into 1-cm sections, which are eluted individually with ethanol (3 ml) for colorimetry. The results obtained with urine from ten normal subjects are tabulated and discussed.

E. C. Apling

1183. **Quantitative evaluation of urinary aldosterone.** C. Matarazzo, M. L. Orabona, R. Chiummo and G. Sacchetti (Ist. Patologia Speciale Medicale, Univ. Bari, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1960, **36** (16), 866–868.—The method described is substantially that due to Romani (*Pr. Méd.*, 1958, No. 37, 837). Half the 24-hr. sample of urine is adjusted to pH 1·5 by the addition of conc. HCl, set aside for 24 hr. to hydrolyse, and then extracted with $CHCl_3$ (3 $\times$ 0·33 vol.). The $CHCl_3$ extract is washed with 0·1N-NaOH (2 $\times$ 0·05 vol.), neutralised with 0·1N-HCl (0·05 vol.), separated, dried over Na_2SO_4 and evaporated to dryness at 50°. The residue is treated by the method of Matarazzo *et al.* (*Anal. Abstr.*, 1961, **8**, 1182). The paper from

2 cm above to 4 cm below the zone corresponding to the cortisone standard is eluted with ethanol (20 ml) and chromatographed on a strip (5 cm × 25 cm) of Whatman No. 1 paper, with system No. 1 of Pechet (*Science*, 1955, **121**, 29). The *R* value for aldosterone, relative to cortisone, is 0·72 to 0·75. Centimetre lengths of the paper are eluted individually with ethanol (3 ml) for spectrophotometry and colorimetry with alkaline blue tetrazolium. Aldosterone exhibits an absorption max. at 285 to 289 mμ. E. C. APLING

1184. **Microchemical detection of characteristic functional groups on the steroid nucleus.** L. R. Axelrod and J. E. Pulliam (Dept. of Physiol. and Biochem., Southwest Foundation for Research and Education, San Antonio, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1200–1202.—Methods are presented for the selective microchemical identification of specific functional groups found in steroid compounds. Most of these identifications can be made with 10 μg or less of the compound per sq. cm of filter-paper test-strip. K. A. PROCTOR

1185. **Two modifications of the colorimetric procedure for determination of serum cholinesterase. Application to Trithion [*S*-(*p*-chlorophenylthio)-methyl *OO*-diethyl phosphorodithioate] and Phosdrin [1-methoxycarbonylprop-1-en-2-yl dimethyl phosphate, 60% technical].** N. R. Rosenthal (Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 737–739.—Cook's method (*Ibid.*, 1954, **37**, 561) has been modified, and the improved procedure used to measure the anticholinesterase activity of Trithion and Phosdrin; the latter substance is about 100 times as active as the former. A. A. ELDRIDGE

1186. **Spectrophotometric titration and ultraviolet-difference spectra of myosin and the meromyosins.** A. Stracher (Dept. of Biochem., State Univ. of New York Coll. of Med., Brooklyn, U.S.A.). *J. Biol. Chem.*, 1960, **235** (8), 2302–2306.—The titration and difference spectra provide evidence that the tyrosyl and tryptophanyl residues of myosin and L-meromyosin are involved in H or hydrophobic bonds. But there is no bonding of this type in the H-meromyosin fragment. J. N. ASHLEY

1187. **Carboxypeptidase B. IV. Purification and characterisation [and determination] of the porcine enzyme.** J. E. Folk, K. A. Piez, W. R. Carroll and J. A. Gladner (Nat. Inst. of Dental Res., Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1960, **235** (8), 2272–2277.—A sensitive and convenient spectrophotometric assay is described. It is based on the difference spectra of hippuric acid relative to hippuryl-L-arginine; the changes in extinction at 254 mμ are measured during hydrolysis of the dipeptide by the enzyme. The rate of hydrolysis is proportional to enzyme concn. J. N. ASHLEY

See also Abstracts—**896**, Automation of enzyme, amino-acid and ammonia determinations. CO_2 in blood serum. Blood glucose *in vivo*. **949**, Sr in biological materials. **1056**, Pyrolysis and gas chromatography of phenylalanine. **1057**, Detection of org. compounds on paper. **1075**, Determination of carbohydrates. **1076**, Chromatography of methylated sugars. **1084**, Detection of cyanoacetic acid. **1087**, Amines in urine. **1088**, EDTA in plasma or serum. **1102**, Use of Keller reaction for indoles and pyrrole. **1206**, Kanamycin in biological fluids. **1266**, Microbiological assay of amino-acids. **1297**, Scintillation counting of ^{14}C compounds. **1312**, **1314**, **1316**, **1317**, **1318**, **1321**, Apparatus and methods for protein electrophoresis. **1315**, Haemoglobin determination. **1327**, I.r. cell for volatile compounds in blood.

Pharmaceutical analysis

1188. **Detection of organic compounds. [II.]** L. Rosenthaler (Seftigenstrasse 40, Bern, Switzerland). *Pharm. Acta Helv.*, 1960, **35** (8), 385–391 (in German).—The following are considered—colour reactions of alcohols, procaine, *o*- and *p*-aminobenzoic acids, ephedrine and amidopyrine; tests for higher alcohols in ethanol and for glyoxylic acid in acetic acid; reactions of aromatic nitro-compounds with ascorbic acid; colour reactions of brucine, hexamine, isoniazid, phenobarbitone, pyridine, thiourea and thiosinamine with aconitic acid and acetic anhydride; and the identification of acetic acid. A. R. ROGERS

1189. **Infra-red analysis of pharmaceuticals. I. Application of the potassium bromide disc technique to some steroids, alkaloids, barbiturates and other drugs.** A. L. Hayden and O. R. Sammul (Div. of Pharm. Chem., Food and Drug Admin., Washington, D.C.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (8), 489–496.—Hand-grinding for 10 min. of soln. of substances with KBr and vibrator-grinding of residues from soln. or recrystallised samples with KBr gave reproducible homogeneous mixtures. After pressing, the discs were heated at 105° for 10 to 20 min. The Beer - Lambert law was obeyed within ±1% by cortisone, hydrocortisone, ethinyloestradiol and ethinyltestosterone over the concn. range 0·05 to 0·4%, and within ±4% by another 7 drugs. Internal standards were not used. Comparison of standard and sample subjected to the same separation procedure, solvent and grinding technique gave results that agreed within ±3% with those obtained by other methods of analysis of a number of pharmaceutical preparations.

II. A study of the cinchona alkaloids in potassium bromide discs. A. L. Hayden and O. R. Sammul. *Ibid.*, 1960, **49** (8), 497–502.—Under certain experimental conditions, anomalous i.r. spectra are obtained for quinine and quinidine. This is thought to be due to the formation of dimorphous and amorphous forms. Cinchonine and cinchonidine do not exhibit significant variations in spectra, and follow the Beer - Lambert law within ±3% at concn. <0·4%. A. R. ROGERS

1190. **Rapid and simple titrimetric method for the determination of alkaloids and potassium with tetraphenylborate and "reversed dead-stop" end-point detection.** C. W. R. Phaf (Farm. Lab., Rijksuniv., Utrecht, Netherlands). *Pharm. Weekbl.*, 1960, **95** (16), 517–535.—A survey is given of methods for the determination of several inorganic and organic bases with tetraphenylborate as a pptg. reagent, and a simple and rapid procedure is proposed whereby interferences are eliminated by isolation of the ppt. *Procedure*—Adjust the sample soln. containing 1 to 50 mg of alkaloid to pH 5 to 7 and add an excess of 0·05N-Na tetraphenylborate. After setting aside the soln. for some time, filter by suction through a G4 filter and wash the ppt. with as little water as possible. Dissolve the ppt. in acetone and titrate with 0·1N- or 0·01N-$AgNO_3$ to the "reversed dead-stop" end-point, with two

identical silver electrodes and applying a potential of 25 mV. Results are given for the determination of K, morphine, papaverine and reserpine, and show satisfactory recovery. M. J. MAURICE

1191. **The determination of papaverine hydrochloride in pharmaceutical products.** C. Maiorovic and Z. Cojocaru (Fac. of Pharm., Cluj, Romania). *Rev. Chim., Bucharest*, 1960, **11** (7), 411–413.—The proposed colorimetric method is based on the solubility in $CHCl_3$ of the ppt. formed by papaverine (**I**) and $Co(SCN)_4(NH_4)_2$ (**II**). *Procedure*—The sample containing between 0·05 and 0·1 g of **I** is dissolved in 5 ml of water, and filtered if necessary, the pH is adjusted to 6 with dil. HCl, and an excess of $\simeq$5 to 8 drops of **II** soln. [3 g of $Co(NO_3)_2$ and 12 g of NH_4SCN dissolved in 10 ml of H_2O] is added. The mixture is extracted successively with 10 ml, 5 ml and 5 ml of $CHCl_3$, and the total extract is diluted to 25 ml with $CHCl_3$. The colour is measured in a colorimeter against a standard treated exactly as the sample or against a soln. of methylene blue. A yellow or orange filter gives greater sensitivity. The method is applicable in the presence of phenobarbitone, lactose, theobromine, ephedrine, codeine phosphate, aspirin or caffeine citrate. Atropine and phenazone interfere. Errors are $\not> \pm 2\%$. H. SHER

1192. **Radio-chromatographic determination of alkaloids in tincture and extract of nux vomica.** M. Šaršúnová, J. Tölgyessy and J. Majer (Regional Control Lab. KSL, Bratislava, Czechoslovakia). *Českosl. Farm.*, 1960, **9** (7), 339–342.—The sample of tincture or of the extract diluted 1 : 100 with 60% ethanol is placed on Whatman No. 1 paper and submitted to chromatography by the descending technique with neutral butanol satd. with H_2O (7 : 3) as solvent. The separated spots are detected by immersing the paper in a soln. of molybdophosphoric acid containing ^{32}P (detailed procedure for the preparation is given), the excess of the reagent is washed out with 2·5% HCl and the chromatogram is dried. The quant. evaluation of the spots is carried out photographically on X-ray film or by means of a Geiger - Müller counter. J. ZÝKA

1193. **Modification of Rumpel's method for the macro-determination of the total alkaloids of ergot.** L. Wichliński (Pharm. Co-op. "Filofarm," Bydgoszcz, Poland). *Farm. Polska*, 1960, **16** (8), 151–153.—A new method, based on that of Rumpel (*Pharmazie*, 1955, **10**, 204) is described. *Procedure*—Place 5 g of powdered ergot (1 mm mesh) in a flask, add 4% (w/v) tartaric acid soln. in aq. methanol (1 : 1) (100 ml) and heat for 20 min. at 40° to 50° with occasional stirring. To the warm soln. add 10% Zn acetate soln. (50 ml) and set aside for 10 min. Filter the upper layer, mix 2·5 ml of the filtrate with 5 ml of dimethylaminobenzaldehyde soln. (B.P. 1958) and after 15 min. measure the extinction in a photometer. Then mix 2·5 ml of filtrate with 5 ml of H_2SO_4 soln. [65 ml of H_2SO_4 (sp. gr. 1·84) mixed with 35 ml of water] and after 1 min. measure the extinction. Deduct this result from that previously determined. As a standard use a soln. of ergotamine in 1% aq. tartaric acid. The complete determination requires 60 min., and the accuracy is within $\pm 5\%$. B. K.

1194. **Analytical investigation of ergot and its alkaloids by the u.v. spectrophotometer.** J. Bayer. *Pharm. Zentralh.*, 1960, **99** (8), 478–484.—Alkaloids of ergot are partially separated and determined as described previously (*cf. Anal. Abstr.*, 1959, **6**, 3156). The powdered ergot is defatted with light petroleum in a Soxhlet apparatus before assay, and the alkaloids are liberated with MgO in the presence of H_2O in the proportion of 1 ml per g of drug. A. R. ROGERS

1195. **The determination of the caffeine content of liquid extract of kola and kola seeds by ion exchange.** W. Kamp (Farm. Lab., Rijksuniv., Utrecht, Netherlands). *Pharm. Weekbl.*, 1960, **95** (15), 481–486.—The usual methods for the determination of caffeine in kola and kola extract have the disadvantage that the caffeine finally isolated is not pure, so that results are high. A preliminary purification by ion exchange is recommended. The liquid extract ($\simeq$3 g) is diluted with 0·1N-HCl (50 ml) and, after filtration, is passed through a column (30 ml) of Dowex 50-X2 (H^+ form). The column is washed first with 0·1N-HCl (250 ml) and then with water (100 ml), and the caffeine is then eluted with acetate buffer (N-acetic acid - 0·1N-ammonium acetate) (300 ml). The first 100 ml of eluate is discarded, the remainder is evaporated to dryness and the residue is dissolved in 2N-aq. NH_3 (50 ml). This soln. is passed through a column of Dowex 1X-2, which has been previously washed with 2N-aq. NH_3, and the caffeine is eluted with 2N-aq. NH_3 (150 ml). The eluate is evaporated to dryness, and the residue weighed. M. J. MAURICE

1196. **Analysis of purine derivatives. II. The bromimetric determination of caffeine, 1-(dihydroxypropyl)theobromine and 7-(dihydroxypropyl)theophylline.** R. Ott and H. Raber (Inst. f. organ. u. pharm. Chem., Univ., Graz, Austria). *Sci. Pharm.*, 1960, **28** (3), 206–215.—To a soln. containing caffeine (**I**) (20 to 200 mg), 1-(2,3-dihydroxypropyl)-theobromine (**II**) (30 to 260 mg) or 7-(2,3-dihydroxypropyl)theophylline (**III**) (30 to 200 mg) in H_2O (30 ml) add 0·1N-$KBrO_3$ (50 ml), 18% aq. KBr soln. (10 ml) and 4N-H_2SO_4 (10 ml), set aside in the dark for 30 min. (for **I** or **II**) or for 5 hr. (for **III**), add a conc. aq. soln. of KI and titrate with 0·1N-$Na_2S_2O_3$. The error is generally $< \pm 1\%$. A. R. ROGERS

1197. **Identification and determination of theophylline in the presence of glucose and sodium salicylate by paper chromatography.** M. Popa and M. Sterescu. *Rev. Chim., Bucharest*, 1960, **11** (7), 420.—The descending method in a butanol - water (2 : 1) system is used. Theophylline is identified as a yellow fluorescent spot at R_F 0·16, while Na salicylate is identified as a blue spot at R_F 0·60. H. SHER

1198. **The assay of leaves of *Digitalis purpurea*.** D. G. Constantinesco, M. Constantinescu, V. Calcandi, J. Ciropol and V. Papa (Inst. de Rech. Pharm. et de Contrôle des Médicaments, Bucharest, Romania). *Ann. Pharm. Franç.*, 1960, **18** (5), 324–328.—Extracts (12) prepared under various conditions from 7 samples of leaves of *Digitalis purpurea* are assayed for toxicity in the guinea-pig by the method used for the preparation of the 3rd International Standard (*Bull. World Hlth Org.*, 1950, No. 2, appendix No. 1), and also by chromatographic analysis of the various glycosides (Kaiser, *Ber. dtsch. chem. Ges.*, 1955, **88**, 556). There is little relation between total glycosides and toxicity, but by multiplying the amount of each individual glycoside by a factor characteristic of its toxicity a total "conventional content" proportional to the

guinea-pig toxicity is obtained. The toxicity depends mainly on the proportions of primary and formylated glycosides, and not on the total glycoside content. E. J. H. BIRCH

1199. **Determination of cardiotonic glycosides with xanthhydrol.** P. Horák (Res. Inst. Med. Plants, Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (7), 1974–1977 (in German).—The effect of heating on the intensity of the colour produced by the reaction between xanthhydrol in glacial acetic acid and cardiotonic glycosides has been studied (*cf.* Pesez, *Ann. Pharm. Franç.*, 1952, **10**, 104). Optimum conditions are—heating at 60° for 60 min., cooling in water at 15° for 5 min., and measurement of the extinction at 530 mμ after 30 min. The colour produced under these conditions is stable for 3 hr. T. R. ANDREW

1200. **A chemical-analytical method for the determination of anthraquinones in the root system of *Rheum palmatum* L.** W. Floret (Pharmakogn. Inst., Univ. Münster/Westf.). *Dtsch. ApothZtg*, 1960, **100** (32), 945–948.—Free anthraquinones are extracted from the drug with benzene. Anthraquinones present as glycosides are hydrolysed by boiling with 0·25% HCl and then extracted with benzene. Rhein, rheinanthranol, physcion (1,8-dihydroxy-3-methoxy-6-methylanthraquinone), chrysophanic acid and rheum- and aloe-emodin are separated by fractional extraction with alkali solutions and determined photometrically. A. G. COOPER

1201. **Separation and determination of the anthraquinones of rhubarb by continuous paper electrophoresis and by counter-current distribution.** G. M. Nano (Ist. Chim. Pharm. e Toxicol., Univ., Turin, Italy). *Pharm. Acta Helv.*, 1960, **35** (8), 451–458 (in French).—The anthraquinones are partially separated by electrophoresis at a potential of 400 V on Whatman No. 1 paper in borate buffer soln. of pH 9·5 at an ionic strength of 0·07, or by counter-current distribution between $CHCl_3$ and 0·4M-borate buffer of pH 9·8, or between toluene and 0·5M-borate buffer of pH 10·1. They are then determined by evaporation and weighing, or by spectrophotometric measurement at 500 to 520 mμ in 10N-NaOH. A. R. ROGERS

1202. **Determination of streptomycin.** V. D. Kartseva, Yu. S. Chekulaeva, V. B. Korchagin and B. P. Bruns. *Antibiotiki*, 1960, **5** (4), 50–53.—It is shown that streptomycin determinations based on the alkaline hydrolysis of streptomycin in the culture fluid, followed by the steam-distillation of maltol and the determination of the maltol in the distillate, give high values; the culture fluid contains impurities that form steam-distillable compounds during alkaline hydrolysis and enter into the $FeCl_3$ reaction. An ion-exchange method is proposed. Dilute mycelium-free culture fluid (10 to 25 ml) to 500 ml and adjust the pH to 8·3 to 8·5 with NaOH soln. Pass the soln. for 15 to 20 min. through a column containing cationite resin KB-1 (Na^+ form) (7 ml) with particle dimensions of 0·1 to 0·25 mm. Elute with 0·5N-H_2SO_4 (50 ml) for 10 min. Neutralise the acid eluate (20 ml) to phenolphthalein and dilute to 25 ml. Determine the streptomycin by the method of Kartseva and Bruns (*Anal. Abstr.*, 1959, **6**, 1503). The results obtained are in good agreement with those of microbiological determinations (diffusion in agar); the difference between results by the two methods is $\not>$4%. K. R. COOK

1203. **Quantitative determination of tetracyclines by circular paper chromatography.** G. Novelli, E. Superti and C. Cattaneo (Lab. Ric., Squibb S.P.A., Roma, Italy). *Farmaco, Ed. Prat.*, 1960, **15** (8), 483–492.—Separation of tetracycline derivatives is achieved by paper chromatography on circles of Whatman No. 1 paper (24 cm diam.), previously buffered at pH 3·5 with McIlvaine buffer and blotted dry, with pyridine - benzene - nitromethane (3:10:20). The sample soln. (0·10 to 0·15 ml, containing 2 to 6 mg per ml) is applied in an arc near the centre of the paper and the solvent is applied to the centre of the disc by means of a wick. Rings of the individual tetracycline derivatives are detected by observation in u.v. light and are dissolved in 0·1N-HCl for determination by spectrophotometry at 355 to 428 mμ. Values of $E^{1\%}_{1\,cm}$ for pure soln. and correction factors for the evaluation of chromatographic eluates are tabulated for tetracycline, 4-epitetracycline, 7-chlortetracycline, 7-chloro-4-epitetracycline, anhydrotetracycline and anhydro-4-epitetracycline. E. C. APLING

1204. **Colorimetric determination of chlortetracycline in turbid culture fluid.** T. N. Laznikova, V. G. Makarevich and T. G. Trofimova (Antibiotics Sci. Res. Inst., Moscow). *Lab. Delo*, 1960, **6** (4), 23–24.—*Procedure for clear culture fluid*—Adjust the pH of the culture fluid from *Actinomyces aureofaciens* with conc. HCl to 2·5 to 3·0 (to tropaeolin OO) and filter off the mycelium after 15 to 20 min. Add 2N-HCl (5 ml) to the filtrate (0·5 to 1 ml), place it in a boiling-water bath for 5 min., allow to cool in a cold-water bath, dilute to 50 ml and measure colorimetrically against a comparison soln. treated similarly (*cf.* Levine *et al.*, *Brit. Abstr. C*, 1950, 18). *Procedure for turbid culture fluid*—Add 5% or 10% trichloroacetic acid soln. (5 ml) to the culture fluid, according to the degree of turbidity, set the mixture aside for 30 to 40 min., filter off the mycelium and then treat the filtrate as already described for culture fluid acidified with HCl. In both cases find the antibiotic concn. from a calibration curve of extinction *vs.* chlortetracycline concn. of known biological activity. K. R. COOK

1205. **Biological determination of fumagillin activity.** V. S. Dmitrieva and S. M. Semenov. *Antibiotiki*, 1960, **5** (4), 46–50.—The method depends on the inhibition by fumagillin of the lysogenic activity of a bacteriophage specific for *Staphylococcus albus* 1623 in a 1·5% agar jelly at pH 6·4 to 6·6 containing 200 mg per 100 ml of pancreatised meat hydrolysate. *Procedure*—Melt the medium and cool it to 48° to 50°, add a suspension of the staphylococcus (5 × 10^7 organisms per ml of medium) and the bacteriophage culture (2% v/v of the medium). Pour the medium into dishes and carry out the cylinder-plate method of assay with fumagillin soln. (10 to 80 units per ml), incubating at 37° for 16 to 20 hr. Use a control containing 20 units per ml. Measure the diameter of the growth zones. The log concn. of antibiotic is directly proportional to the diameter of the growth zone, which represents the limit of linear diffusion of the antibiotic. The fumagillin activity is determined by means of tables. The accuracy of the method is within ±15%. K. R. COOK

1206. **Chemical method for the determination of kanamycin in biological media.** G. Moustardier, C. Dulong de Rosnay, J. Dufau-Casanabe, P. du Pasquier and J. Latrille (Lab. de Bactériol., Fac.

de Méd. et de Pharm., Bordeaux). *Ann. Inst. Pasteur*, 1960, **99** (3), 447–449.—*Procedure*—Kanamycin soln. (0·5 ml) is treated with H_3BO_3 - H_2SO_4 soln. [H_2SO_4 (1650 g per litre) containing 5% of H_3BO_3] (4 ml) and immersed in a boiling-water bath for 10 min. After cooling, satd. carbazole soln. in 95% ethanol (0·5 ml) is added and the mixture is replaced in the bath for a further 5 min. A violet colour develops, which is read against a reagent blank; 10 µg of kanamycin gives an extinction of 0·15, and the precision is ±5%. For biological media, the kanamycin is pptd. with calcium phosphate soln. (1% of $CaCl_2$ and 4% of Na_2HPO_4 in water, dissolved by the addition of a few drops of HCl) in the presence of acetone. Urine is treated with one-tenth of its vol. of conc. H_2SO_4, then hydrolysed for 30 min. in a boiling-water bath and filtered if necessary. The filtrate (1·1 ml) is treated with phosphate soln. (0·1 ml) and acetone (5 ml), and centrifuged. The supernatant liquid is decanted off and the residue is freed from acetone by evaporation, and treated with 1 ml of water; 0·5 ml of the soln. is taken for measurement. Culture media are treated similarly. Blood serum is pptd. with an equal vol. of 20% trichloroacetic acid soln., and the filtrate is treated as for urine, taking 2·2 ml of hydrolysate, 0·2 ml of phosphate soln. and 10 ml of acetone. The residue is treated with 0·5 ml of water, and this soln. is measured directly. Serum not containing kanamycin gives a blank extinction of ≃0·20. R. E. E.

1207. **Colorimetric determination of nystatin.** W. H. Unterman (Antibiotics Factory, Iaşi, Romania). *Rev. Chim., Bucharest*, 1960, **11** (7), 419–420.—The method is based on the colour produced with molybdoarsenate (**I**). The sample is dissolved in dimethylformamide to give a concn. of between 50 and 100 µg per ml, and a 2-ml aliquot is treated with 2 ml of 6N-H_2SO_4 and 1 ml of **I** soln. The mixture is boiled for 15 min., cooled, and diluted to 25 ml, and the extinction is measured in a Pulfrich photometer with a S42 filter. A standard curve is prepared from a soln. of known concn. The sensitivity of the method is 20 µg per ml. H. Sher

1208. **Psicofuranine: correlation of assay methods in acid degradation studies.** E. R. Garrett and L. J. Hanka (The Upjohn Co., Kalamazoo, Mich., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (8), 526–529.—Adenine, a product of the acid-catalysed hydrolysis of psicofuranine, appears to reverse the microbiological activity of psicofuranine in the plate-disc method against *Staphylococcus aureus* (*cf.* Hanka *et al.*, *Antibiot. & Chemother.*, 1959, **9**, 432), so that standard curves must be prepared with the same amount of adenine as the material to be assayed. When allowance is made for this, the results from the chemical assays (*cf.* Forist *et al.*, *Anal. Abstr.*, 1960, **7**, 3406; Forist, *Ibid.*, 1960, **7**, 2937) and microbiological assays agree. A. R. Rogers

1209. **Identification of barbiturates from their infra-red spectra.** B. Cleverley (Dominion Lab., D.S.I.R., Wellington, New Zealand). *Analyst*, 1960, **85**, 582–587.—The barbiturate to be identified (≃0·7 to 0·8 mg) is ground by hand for 5 min. with ≃300 mg of KBr of i.r. quality. To prepare the disc, the mixture is placed in an oven maintained at 10° above the determined m.p. of the sample, and after 30 min. is removed and rapidly cooled to room temp., and its spectrum recorded between 4000 and 650 cm^{-1}. Twenty barbiturates were studied, and when the spectrum obtained by this method is not available in the literature the graphs are given. The method is suitable for toxicological samples, since only small amounts are required. A. O. Jones

1210. **Rapid chromatographic detection of barbiturates.** M. Ledvina (I Inst. Med. and Forensic Chem., Prague). *Českosl. Farm.*, 1960, **9** (7), 333–335.—The soln. of the sample is placed on Whatman No. 1 paper that has been impregnated with a 50% soln. of formamide in ethanol and dried. The chromatogram is developed with $CHCl_3$ or CCl_4 or their mixture and after 2 to 3 hr. is dried at 90° to 100°; the detection is as previously described (*Anal. Abstr.*, 1956, **3**, 1148). The chromatographic procedure can be accelerated by carrying out the development at 30°, 40° or 50° with n-pentanol - conc. aq. NH_3 (2:1). J. Zýka

1211. **Detection of barbiturates in paper chromatography.** M. Ledvina and J. Turek (Inst. of Med. and Forensic Chem., Charles' Univ., Prague). *Českosl. Farm.*, 1960, **9** (7), 354–356.—The detection of barbiturates in u.v. light is more sensitive than by the use of HgI or Zwikker's reagent. Mixtures of allobarbitone, aprobarbital, phenobarbitone and barbitone were separated on S. & S. 2043b or Whatman No. 1 paper, with butanol - aq. NH_3 (1:1). J. Zýka

1212. **Identification of sulphonamides.** A. Goudswaard (Rijks-Inst. Pharm. Therap. Onderz., Leiden, Netherlands). *Pharm. Weekbl.*, 1960, **95** (15), 487–489.—Tests with Cu^{II} acetate soln. are described for the detection of sulphadiazine, sulphadimidine and sulphamerazine in mixtures with other sulphonamides or with each other. M. J. Maurice

1213. **Micro-determination of the molecular weight of some synthetic antihistamines as a criterion for their identification.** S. Bruno (Inst. Pharm. Chem. and Toxicol., Univ., Bari, Italy). *Farmaco, Ed. Prat.*, 1960, **15** (9), 543–546.—The principle that the visual or u.v. absorptions of isolated chromophores are not affected by that of the remainder of the molecule leads to the relationship $E^{1\,\%}_{1\,cm} = 10\epsilon/M$, where E is the observed extinction, ϵ is the mol. extinction coeff. and M is the mol. wt. This relationship, applied to picrates of bases such as antihistamines, leads to the formula $M = 13{,}440.C.n/\log(I_0/I)$, where M is the mol. wt. of the picrate, C the concn. in g per litre and n the mol. ratio of picric acid to base; 13,440 is the value of ϵ_{380} for the picric chromophore. Picrates of antihistamines were prepared by dissolving 0·2 g of the salt in 20 to 25 ml of 95% ethanol, adding 20 ml of 1·1% aq. picric acid, warming till dissolution was complete, allowing to crystallise and re-crystallising from ethanol or acetone; 1 to 2 mg of the product was dissolved in 100 ml of ethanol and the extinction was measured at 380 mµ in a Beckman DU spectrophotometer. The results for 10 antihistamines of mol. wt. from 484 to 743 differed from the theoretical by amounts ranging from −1·04 to +0·79%. J. I. M. Jones

1214. **New method for the detection of fractional p.p.m.-quantities of diethylstilboestrol [stilboestrol].** J. M. Goodyear and N. R. Jenkinson (Control Div., Eli Lilly and Co., Indianapolis, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1203–1204.—The method described depends on the u.v. irradiation of stilboestrol in re-distilled ethanol and measurement

of the fluorescence produced. The method is highly sensitive and can be used to distinguish the natural and other synthetic oestrogens from stilboestrol, with the exception of dienoestrol which is about 25% as active as stilboestrol in the test described.
K. A. PROCTOR

1215. **Determination of *p*-acetamidophenol (acetaminophen N. F. XI) in some pharmaceutical formulations.** K. T. Koshy and J. L. Lach (Coll. of Pharm., Univ., Iowa City). *Drug Standards*, 1960, **28** (4), 85–87.—Eight pharmaceutical preparations of *p*-acetamidophenol (**I**) have been analysed spectrophotometrically and by the colorimetric method of Greenberg and Lester (*J. Pharmacol.*, 1946, **88**, 87). Both methods are satisfactory, except that the results by the spectrophotometric method are high in the presence of acetylsalicylic acid. *Procedure*—Pass a soln. (10 ml, containing $\simeq$1 mg of **I**) through a column of Amberlite IR-120 (H^+ form) (0·5 g) and elute with H_2O to produce 100 ml. For the spectrophotometric determination, measure the extinction at 244 mμ. For the colorimetric determination, hydrolyse a 10-ml aliquot with conc. HCl (2 ml), add a fresh 10% soln. of 1-naphthol in ethanol and 30% NaOH soln. and measure the extinction at 590 mμ.
A. R. ROGERS

1216. **Determination of 6-(4-carboxybutylmercapto)purine (Buthiopurine).** F. Jančik, B. Kakáč and B. Budešínský (Res. Inst. Pharm. and Biochem., Prague). *Českosl. Farm.*, 1960, **9** (7), 329–333.—*Argentimetric method*—Dissolve the sample (0·3 to 0·4 g) in 2N-HNO_3 (25 ml), add 0·1N-$AgNO_3$ (20 ml), mix, dilute with H_2O to 100 ml, filter and determine the excess of Ag with 0·1N-KSCN in the usual way. A direct argentimetric determination can be performed potentiometrically. *Alkalimetric method*—Neutralise dimethylformamide (10 ml) with 0·1N-K methoxide, with azo violet as indicator. Add the sample (40 to 120 mg) and titrate with 0·1N-K methoxide till the colour again turns blue. The potentiometric determination also yields good results; two potential changes indicate the neutralisation of the carboxyl and the imino-groups. *Spectrophotometric determination*—Dissolve the sample (100 mg) in HCl (1:7) (40 ml), dilute with H_2O to 100 ml and dilute 5 ml of this soln. to 100 ml with H_2O. Dilute 5 ml of this soln. to 25 ml with 0·1N-HCl. Measure the extinction at 209 mμ, and compare with a calibration curve or use a formula for the calculation of the result. With tablets, **I** must be first extracted with HCl (1:1) and the undissolved residue filtered off.
J. ZÝKA

1217. **Ultra-violet spectrophotometric method for the determination of picrate, styphnate and picrolonate in *S*-alkyl-*N*-phenylthiuronium salts.** J. Thomas and W. A. Baker (Dept. of Pharm., Univ., Manchester). *J. Pharm. Pharmacol.*, 1960, **12** (8), 466–472.—Prepare a soln. of *S*-alkyl-*N*-phenylthiuronium picrate (0·001 to 0·004%), styphnate (0·001 to 0·004%) or picrolonate (0·001 to 0·0025%) in 95% ethanol and measure the extinction in a 1-cm cell at 385 mμ (for picrate), 400 mμ (styphnate) or 350 mμ (picrolonate). *N*-Phenylthiourea does not absorb light in the region from 350 to 400 mμ. The method is accurate to $\pm$1%.
A. R. ROGERS

1218. **Complexometric estimation of zinc in pharmaceuticals.** P. M. Parikh, D. J. Vadodaria and S. P. Mukherji (Zandu Pharmaceutical Works Ltd., Bombay). *Indian J. Pharm.*, 1960, **22** (9), 229–231.—Results for the assay of ZnO, $ZnSO_4$ and $ZnCl_2$ and their pharmaceutical preparations by the method described are in good agreement with those by the methods of the B.P., the B.P.C. and the International Pharmacopoeia. *Procedure*—Prepare an aq. soln. of the sample (50 ml containing $\simeq$50 mg of Zn). Remove dyes by adsorption on activated charcoal. Remove ointment bases or oil by extraction with $CHCl_3$. Add aq. NH_3 until the soln. is slightly alkaline, then add 5 ml of buffer soln. [dissolve NH_4Cl (33·8 g) in 20% aq. NH_3 (425 ml), add $MgSO_4.7H_2O$ (0·308 g) and 0·1N-EDTA (disodium salt) (25 ml) and dilute with H_2O to 500 ml] and titrate with 0·1N-EDTA (disodium salt) with Solochrome black WDFA (C.I. Mordant Black 11) as indicator.
A. R. ROGERS

1219. **The determination of chloramine T in the presence of formaldehyde.** R. Jensen, S. Garrin and F. Tayeau (Lab. de Chim. Biol. et Méd., Fac. de Méd., Bordeaux, France). *Bull. Soc. Chim. France*, 1960, (5), 975–977.—The titration of chloramine T according to the method of the French Codex (in which KI is added before the acetic acid, followed by titration of the liberated iodine with $Na_2S_2O_3$) gives low and irregular results in the presence of formaldehyde. This is shown to be due to the liberation of KOH by the reaction of KI with neutral chloramine T, which permits the liberated iodine to oxidise the formaldehyde in the alkaline soln. It is therefore proposed that the Codex method should be modified by adding the acetic acid before adding the KI. This method is equally applicable to pure chloramine-T soln.
E. J. H. BIRCH

See also Abstracts—**896**, Automatic assay of antibiotics. Continuous analysis of penicillin in fermentation media. **1056**, Gas chromatography of drugs by pyrolysis and gas chromatography. **1102**, Keller reaction for lysergic acid. **1118**, Eugenol in clove oil. **1146**, Barbiturates in blood. **1147**, Phenothiazine derivatives in urine. **1148**, Laevomycetin in biol. liquids. **1155**, Glyoxylic acid reaction for indoles. **1156**, Gentisic acid in biol. fluids. **1246**, Vitamin-B group in multivitamin preparations. **1329**, Determination of sulphonamides.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

1220. **Application of the cathode-ray polarograph in food analysis.** J. S. Hetman (Southern Analytical Ltd., Camberley, Surrey, England). *Lab. Practice*, 1960, **9** (8), 563–567.—A lecture.

1221. **Determination of the raffinose content of molasses by a paper-chromatographic method.** R. I. Gol'dfarb and P. L. Danilenko. *Sakhar. Prom.*, 1960, (5), 21–23.—A clarified molasses soln. is inverted with acid (whereby raffinose is converted into melibiose), neutralised with NaOH and adjusted to a concn. of 13 g of molasses in 100 ml. Aliquots (0·05 ml) are developed with n-butanol - acetic acid - water (4:1:5) (for descending chromatography only) or n-butanol - ethyl acetate - water (7:1:2) as solvent. The development takes 40 to 48 hr. and the spots are revealed with a soln. containing 0·5 g of benzidine hydrochloride in 80 ml of ethanol with 5 ml of 80% trichloroacetic acid and

10 ml of glacial acetic acid. Strips containing the melibiose are cut out and extracted with 50 to 100 ml of water for 30 to 45 min. at 40°. The extract is evaporated to 5 ml and a 1-ml aliquot is heated with 1 ml of copper reagent, comprising equal quantities of (*i*) 13 g of copper sulphate in 1 litre and (*ii*) a mixture of 50 g of $NaHCO_3$ and 40 g of Na_2CO_3 in 700 ml, 36·8 g of K oxalate in 120 ml, and 24 g of K Na tartrate in 100 ml of water. The reaction mixture is cooled and 2 ml of water and 1 ml of Nelson's reagent (containing 25 g of ammonium molybdate in 450 ml of water, 21 ml of conc. H_2SO_4 and 3 g of sodium arsenate in 25 ml of water) are added. The stable blue complex formed with cuprous oxide is measured colorimetrically. The results agree with those obtained by the double enzyme method to within $\pm10\%$.

SUGAR IND. ABSTR.

1222. **Improved colorimetric method for determining ferrocyanide ion, and its application to molasses.** J. R. Marier and D. S. Clark (Div. of Applied Biology, National Res. Council, Ottawa, Canada). *Analyst*, 1960, **85**, 574–579.—An aliquot ($\not>$5 ml) of sample soln., diluted standard soln. [5 to 80 μg of $Fe(CN)_6^{4-}$] or water (blank) is placed in a colorimeter tube, its vol. is adjusted to 5 ml, 2 ml of 50% citric acid soln. is added and then 1 ml of $FeCl_3$ soln. (0·6% w/v in 0·1N-HCl). After 60 to 90 min. at room temp. the extinction is measured at 690 mμ. To correct for the colour of molasses, a duplicate aliquot of sample is treated similarly, but with 1 ml of water replacing the $FeCl_3$ soln. The extinction, measured against a normally prepared reagent blank, is deducted from that of the test soln. When the method is used for the determination of residual $Fe(CN)_6^{4-}$ in beet molasses the presence of up to 3 ml of diluted molasses (25% dilution) has no adverse effect. The results of recovery experiments averaged $100·3 \pm 3·2\%$, and were not significantly affected if the sample had undergone fermentation treatment.

A. O. JONES

1223. **The accuracy of the Babcock test for the determination of fat in milk.** J. H. Labuschagne and K. F. Vogt (Div. Animal Husbandry and Dairying, Pretoria). *S. Afr. J. Agric. Sci.*, 1960, **3** (2), 305.—The relationship between results by the Röse-Gottlieb method (R.G.) and those by the Babcock method (B) for 89 samples is given by the equation B = 1·020 R.G. − 0·074. Results for the two methods agree at a fat level of 3·70%. The equation obtained by Labuschagne and Vogt (*Ibid.*, 1960, **3**, 83) for the Gerber method agrees closely with that given above. I. DICKINSON

1224. **The TeSa test: new rapid method for fat in milk and milk products.** B. B. Anderson, D. W. Bailey, J. C. Ash and B. Jaquith (Anderson Lab., Inc., Fort Worth, Tex.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (2), 399–405.—A method is described in which the use of H_2SO_4 is eliminated. The reagent contains urea, Na_2CO_3, EDTA, Na_2HPO_4 and polyoxyethylene esters of mixed fatty and resin acids. Special bottles are used, the milk being mixed with the reagent and heated in boiling water for 10 to 12 min. The vol. of fat is read on a graduated scale. Methods are described for homogenised milk, cream and other milk products. N. E.

1225. **Comparison of the TeSa test with the Mojonnier or Röse-Gottlieb test and the Babcock test on raw milk.** R. G. Washburn (Dept. Dairy Sci., Ohio Agric. Exp. Sta., Wooster, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 746–748.—Results of comparative collaborative tests were in excellent agreement. The TeSa fat test for fresh milk (*cf.* Anderson *et al.*, *Anal. Abstr.*, 1961, **8**, 1224) gives reliable results. A. A. ELDRIDGE

1226. **Use of methylal for the determination of fat in milk.** P. Navellier and R. Brunin, with M. Surre (Lab. Municipal, Paris, France). *Ann. Falsif.*, 1960, **53**, 379–390.—Several methods for the extraction of fat from milk are compared, and the most effective is shown to be a continuous extraction by an azeotrope of methylal and methanol. *Procedure*—A mixture of methylal and methanol (4:1) is placed in the boiler of a B.B.S. macro extractor for use with light solvents, and 10 ml of milk is placed in the extraction tube. The milk is extracted for 8 hr. at a rate of 1 drop of solvent per sec. from the condenser. The solvent is recovered and the last traces are removed at 40° to 45°. The residue is extracted with $CHCl_3$ or light petroleum several times, collected in a sintered glass crucible, and, after removal of the solvent on a water bath, weighed. A similar method for powdered milk is described. The chief advantage of the method is that it can be used for milk that has been preserved with formaldehyde. E. J. H. BIRCH

1227. **Determination of iodine-131, caesium-137 and barium-140 in fluid milk by gamma-spectroscopy.** G. R. Hagee, G. J. Karches and A. S. Goldin (U.S. Dept. of Health, Education and Welfare, Robert A. Taft San. Engng Center, Cincinnati, Ohio, U.S.A.). *Talanta*, 1960, **5** (1), 36–43.—The nuclides present together in milk can be determined individually in concn. down to 10 pC per litre with an accuracy within $\pm10\%$ at concn. >100 pC per litre. The milk (3·5 litres) is placed in an aluminium beaker in a scintillation spectrometer, and the spectrum is recorded over the energy range 0 to 1·9 MeV in 95 20-KeV channels. The nuclides are determined by means of four simultaneous equations. No preparation of the sample is required. The sensitivity is $\simeq$10 pC per litre, and the accuracy 5 to 10 pC per litre at nuclide concn. of 50 to 100 pC per litre. G. BURGER

1228. **Detection of β-carotene in orange juice.** Sunkist Growers (Ontario, Calif., U.S.A.). *Fruchtsaft-Ind.*, 1960, **5** (5), 181–182.—The method whereby the ratio of β-carotene to xanthophylls is determined requires overnight saponification. This can be avoided by the simple method described, in which the artificial emulsion of the added pigment is broken by centrifuging. *Procedure*—The orange juice is diluted if necessary to 12° Brix, and a 50-ml aliquot is centrifuged at 3000 r.p.m. for 15 min. Light petroleum (boiling-range 65° to 110°) is added in such a way that only the drops floating on the surface are dissolved. A yellow colour of the light petroleum soln. indicates the presence of added β-carotene. The solvent layer is removed by pipette and the absorption spectrum between 400 and 500 mμ is recorded and compared with that of β-carotene in light petroleum. I. DICKINSON

1229. **Determination of ethanol in fruit juices and aroma concentrates.** G. Senn (E. Brunner Lab., Steinmaur, Switzerland). *Fruchtsaft-Ind.*, 1960, **5** (5), 165–173.—Available methods are discussed, and a rapid method suitable for routine analyses is described. Interfering carbonyl compounds are destroyed with HgO. *Procedure*—Boil the fruit juice or essence (100 ml) under reflux with HgO

(0·5 g) for 1 hr., then distil 80 ml into a 100-ml flask and make up to volume. Determine the ethanol content either by pyknometer or, after oxidation with chromic acid, iodimetrically. (68 references.) I. DICKINSON

1230. **Use of gas chromatography in measuring the ethylene production of stored apples.** D. F. Meigh (Ditton Lab., Larkfield, Maidstone, Kent, England). *J. Sci. Food Agric.*, 1960, **11** (7), 381–385.—The ethylene produced was determined by using a special flame ionisation detector and a large column to separate the gas from interfering substances. B. HEINING

1231. **Separation of organic and inorganic acid anions in filtered tomato purée by partition chromatography.** D. B. Bradley (Agric. Res. Dept., Campbell Soup Co., Riverton, N.J., U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 232–234.—Cations in filtered tomato purée were removed by passage through the cation-exchange resin Dowex-50. The acids in the percolate were then separated on a silicic acid column, and eluted with a series of solvents containing increasing amounts of n-butanol in $CHCl_3$, with methanol as the final solvent. The acids recovered accounted for 94% of the total acidity; acetic, lactic, fumaric, malic, pyrrolidonecarboxylic (pyroglutamic), citric, phosphoric, hydrochloric, sulphuric and galacturonic acids were identified. Differences in relative concn. were observed in different samples of purée. The major constituent was citric acid, which accounted for 51·5 to 55·5% of the total acidity. M. D. ANDERSON

1232. **Analysis of vanilla extracts. I. Organic acid determination.** J. H. Sullivan, W. A. Voelker and W. H. Stahl (McCormick & Co. Inc., Baltimore, Md., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (2), 601–605.—Palmer's method (*Conn. Agric. Exp. Sta. Bull.*, 1955, 589) for the analysis of organic acids in tobacco is, when suitably modified, acceptable for the determination of organic acids in vanilla extract. The procedure, which is described, involves gradient elution chromatography on a Dowex ion-exchange resin, followed by titration of the eluate after removal of formic acid by volatilisation. Recoveries range from 97% to 103%. Differences between *Vanilla planifolia* and *V. tahitensis* are discussed. The method permits the calculation of total bean solids used in the preparation of the extract.

II. Preparation and reproduction of two-dimensional fluorescence chromatograms. W. H. Stahl, W. A. Voelker and J. H. Sullivan. *Ibid.*, 1960, **43** (3), 606–610.—Spots of vanilla extract on chromatograph paper are developed in two directions at right angles with aq. ethanolic $KHCO_3$ and with aq. isopropyl or aq. t-butyl alcohol, respectively. The chromatograms are then photographed in colour while exposed to ultra-violet light. The chromatograms themselves fade after a time. The fluorescence pattern is a series of separate spots, and sophistication, *e.g.*, with cascara, liquorice or yarrow, can thereby be detected. Colour photographs are reproduced. A. A. ELDRIDGE

1233. **Determination of nitrate in meat and meat products.** W. A. Landmann, M. Saeed, K. Pih and D. M. Doty (Amer. Meat Inst. Foundation, Univ. Chicago, Ill., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 531–535.—Conditions for the development of the nitrate - brucine colour reaction have been studied, and a procedure suitable for the direct determination of nitrate in meat and meat products has been evolved. Nitrite is removed by addition of urea. Chloride, ascorbic acid or isoascorbic acid did not interfere under the specified conditions. For nitrate added to fresh or cured meat, recoveries of 95 to 110% are reported. A. A. ELDRIDGE

1234. **Indirect polarographic determination of nitrates in food products.** E. Davídková and J. Davídek (Centr. Res. Inst. Food Industry, Prague). *Průmysl Potravin*, 1960, **11** (7), 385–387.—Nitrates react with 2,4-xylenol yielding the yellowish-red 6-nitro-2,4-xylenol, the polarographic wave of which can be measured in 0·4M-NaOH ($E_{\frac{1}{2}} = -0·82$ V). *Procedure*—To the soln. of NO_3^- (5 ml) (100 to 500 μg) add H_2SO_4 (85%) (20 ml) satd. with Ag_2SO_4, a soln. of 2,4-xylenol in acetone (2%) (1 ml) and, after 20 min., add H_2O, and distil off about 45 ml into a 50-ml flask containing 4M-NaOH (5 ml). Add gelatin soln. (0·5%) (0·5 ml) and register the polarographic wave. The interference of nitrites is avoided by converting them into methyl nitrite and removal by distillation. Proteins (*e.g.*, in steamed food products) are removed by pptn. with a soln. of tungstosilicic acid (20%) (9 ml). J. ZÝKA

1235. **Complexometric determination of antioxidants. II. New semi-micro complexometric method for the determination of nordihydroguaiaretic acid (NDGA) in lard.** B. A. J. Sedláček (Food Research Inst., Prague). *Fette, Seif., Anstrichmitt.*, 1960, **62** (8), 669–672.—Silver nitrate is reduced by NDGA, especially in a soln. in HNO_3 that is buffered with Na acetate. After dissolution of the resulting silver in HNO_3 and neutralisation with aq. NH_3, the soln. is treated with buffer, $K_2[Ni(CN)_4]$ soln., EDTA and Eriochrome black T; the Ni^{2+} displaced by the Ag^+ react with EDTA, the excess of which is titrated with $ZnSO_4$ soln. Results show this direct method to be preferable to an indirect method. Various methods for extracting the antioxidant from lard were investigated, and extraction with purified methanol (50%) is recommended. As $AgNO_3$ is reduced by antioxidants other than NDGA, this method cannot be used for the determination of NDGA in mixtures. I. DICKINSON

1236. **Chromatographic separation and ultraviolet spectrophotometric determination of benzoic acid, *m*-hydroxybenzoic acid, *p*-hydroxybenzoic acid and salicylic acid in mixtures.** H. C. Van Dame (Food and Drug Admin., Dept. of Health, Education and Welfare, Buffalo, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 593–594.—The acids named, extracted from food, may be separated chromatographically on a column of silicic acid, with benzene, and subsequently a mixture of butanol and benzene, as solvent. The results of titration of the eluted fractions should be checked by spectrophotometric analysis. A. A. ELDRIDGE

1237. **Colorimetric micro-determination of 1-chloro-2-nitrobenzene in pineapple.** H. Y. Young (Pineapple Res. Inst. of Hawaii, Honolulu). *J. Agric. Food Chem.*, 1960, **8** (3), 213–214.—1-Chloro-2-nitrobenzene is determined in pineapple tissue by extracting with benzene, freeing the extract from pigments on an Attapulgus clay - Celite column, removing the solvent by evaporating in a covered beaker to 10 ml, reducing in the presence of the residual benzene, diazotising, coupling with *N*-1-naphthylethylenediamine, and measuring the

colour at 540 mμ. The mean recovery of added herbicide was 93%, and 0·01 p.p.m. may be determined. M. D. ANDERSON

1238. **Detection and determination of biphenyl and o-phenylphenol in concentrated orange juice by gas chromatography.** R. Thomas (Min. of Agric., Fisheries and Food, Horseferry Road, London, England). *Analyst*, 1960, **85**, 551–556.—The conc. orange juice (1 ml) is diluted with water (4 ml), 1 ml of the internal standard (a soln. of thymol in methanol diluted with water so that 1 ml ≡ 2 μg of thymol) is added and the mixture is steam-distilled. A 1-ml portion of the distillate is shaken with 0·5 ml of $CHCl_3$ and, by a procedure described, an extract in $CHCl_3$ free from water is obtained. This extract is mixed with some of the column packing (20% w/w of Embophase silicone oil on 100 to 200-mesh Celite), the $CHCl_3$ is allowed to evaporate and the powder is added to the column with only momentary disconnection of the gas inlet. The chromatogram is then run and the areas beneath the peaks corresponding to the components are measured. The experimental error at a probability of 95% is 0·04 p.p.m. for biphenyl, and +0·4 to −0·8 p.p.m. for *o*-phenylphenol, at concn. of 1 to 10 p.p.m. A. O. JONES

1239. **The determination of small amounts of DDT in flour and other foodstuffs.** Report by the DDT Panel set up jointly by the Scientific Subcommittee of the Interdepartmental Advisory Committee on Poisonous Substances used in Agriculture and Food Storage, The Analytical Methods Committee of the Society for Analytical Chemistry and The Association of British Manufacturers of Agricultural Chemicals. *Analyst*, 1960, **85**, 600–606.—The extract of the sample in n-hexane is treated with conc. H_2SO_4 and fuming H_2SO_4, and after 30 min. the organic layer is applied to a column of Celite 545 mixed with the H_2SO_4 mixture and hexane. The extraction of the aq. liquid with hexane is repeated and the combined percolates are reduced in vol. to a few ml in a simplified form of the Kuderna - Danish evaporator. The liquid is then washed with hexane or light petroleum on to a column of silica gel and the solvent. The column is washed with the solvent, drained under pressure and eluted with ethyl ether. The eluate with a few drops of propane-1,2-diol is reduced in vol. to a few ml in the evaporator and the residual solvent is removed at 40°. The residue is heated in a steam bath for 30 min. and then with 2 ml of fuming HNO_3 and H_2SO_4 for 30 min. The nitrated product, diluted with water, is extracted with light petroleum - benzene (2:1), and the separated organic layer is washed with KOH soln. and then with satd. NaCl soln. The residue from evaporation of an aliquot is dissolved in 1 ml of benzene, ethanolic KOH is added and the extinction of the blue liquid is measured at 600 mμ and referred to a calibration graph prepared by nitration of *pp'*-DDT. A. O. JONES

1240. **Determination of heptachlor epoxide in fat and milk.** C. F. Meyer, M. A. Malina and P. B. Polen (Velsicol Chemical Corp., Chicago, Ill., U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 183–186.—Dairy cows fed with forage containing residues of heptachlor convert it into the epoxide, which is stored in fatty tissue, and excreted in milk. The method described for determining heptachlor epoxide is based on that proposed for residues in lucerne. The separated fat is mixed with anhyd. Na_2SO_4, ground, and extracted with pentane. Solvent is removed, and the fat is saponified by acid if both heptachlor and the epoxide are to be determined, or by alkali for the epoxide only. The insecticide is then extracted into pentane, and separated by chromatography on a column of Florex, heptachlor being eluted with pentane, and the epoxide with pentane containing 2% of ether. Heptachlor is determined by the method of Ordas *et al.* (*Ibid.*, 1956, **4**, 444), and the epoxide by reaction with Polen - Silverman reagent (*Brit. Abstr. C*, 1952, 403), modified by dilution with benzene, and spectrophotometric determination of the coloured product at 410 mμ. Correction for the relatively large background absorption is necessary. The presence of much heptachlor affects the values for the epoxide, and necessitates a further correction. M. D. ANDERSON

1241. **Identification of pesticide residues in extracts of fruit, vegetables and animal fats. III. Metabolites of chlorinated hydrocarbon pesticides in animal depot fat.** W. P. McKinley and H. C. Grice (Food and Drug Lab., Dept. of Nat. Health and Welfare, Ottawa, Canada). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 725–731.—Rats were fed on diets containing Kelthane [2,2,2,-trichloro-1,1-di-(*p*-chlorophenyl)ethanol], *pp'*-DDT, *op'*-DDT, Rhothane [1,1-dichloro-2,2-di-(*p*-chlorophenyl)ethane] or chlorobenzilate. Extracts of abdominal fat in CCl_4 were prepared, and the extracted fat was partitioned between methyl cyanide and n-hexane and chromatographed. McKinley and Mahon's method (*Anal. Abstr.*, 1960, **7**, 3020) was employed for detection of the pesticides. The substances so identified are listed for each pesticide ingested. Chlorobenzilate does not appear to be stored in depot fat, and *op'*-DDT is stored only to a slight extent. A. A. ELDRIDGE

1242. **Conductimetric analysis of hop bitter substances [in beer].** J. R. Hudson and A. H. Cooper (Brewing Ind. Res. Foundation, Nutfield, Surrey, England). *J. Inst. Brewing*, 1960, **66** (4), 298–301.—A critical survey of published procedures is given, and the development of the following method is described. *Procedure*—De-gassed beer (100 ml) is placed in a long tube (55 cm × 2 cm), treated with 6N-HCl (15 ml) and pepsin (1:2500, approx. 0·2 g), mixed, and set aside for 30 min. Light petroleum (50 ml, boiling-range 40° to 60°) is then added and the bitter substances are extracted by rocking the stoppered tube. The petroleum phase is then separated, washed with water and then with acidified methanol, dried with anhyd. $MgSO_4$ and evaporated to ≃5 ml under reduced pressure. The residue is diluted to 50 ml with anhyd. methanol, and titrated conductimetrically with 0·02M-methanolic Ni acetate. The graph of meter readings *vs.* vol. of titrant added gives two straight lines. On the assumption that the mean mol. wt. of the isohumulones in beer is 355, the concn. (mg per litre) of bitter substances is then (ml of titrant × 710 × 10)/500. The results are generally higher by ≃3 mg per litre than those given by the second rapid u.v. spectrophotometric method of Rigby and Bethune (*Anal. Abstr.*, 1956, **3**, 250). R. E. E.

1243. **Determination of diene conjugation as an evaluation of antioxidants.** Tyo-taro Tukamoto (Nagoya City Univ. Hospital, Nagoya, Japan). *Pharm. Acta Helv.*, 1960, **35** (8), 431–441 (in English).—The change in the rate of formation of

conjugated dienes in the air-oxidation of unsaturated fatty acids and natural fats and oils is suggested as a measure of the activity of antioxidants. The results agree with those of peroxide value and relative viscosity measurements. *Procedure*—Heat the sample containing the antioxidant in a boiling-water bath and pass O at a rate of 14 ml per min. Once an hour, for 5 hr., remove a 10-mg aliquot, dilute it to 60 ml with ethanol and measure the u.v. absorption at 234 mμ. A. R. ROGERS

1244. **Use of radioactive isotopes for the analysis of vegetable oils.** M. Jáky and K. Kaffka (Fettforschungsinst., Budapest). *Fette, Seif., Anstrichmitt.*, 1960, **62** (8), 682–687.—The accuracy of the radiometric determination of the iodine value with ^{131}I has been confirmed. Experiments are described and results given for the paper-chromatographic analysis of fatty acids in which the acids were converted into silver and mercury soaps labelled with the radioactive isotopes ^{110}Ag and ^{203}Hg. I. DICKINSON

1245. **New spectrophotometric method for the determination of vitamin A. II. Measurement of the blue colour.** I. M. Jakovljevic (Eli Lilly and Co., Indianapolis, Ind., U.S.A.). *Pharm. Weekbl.*, 1960, **95** (17), 549–560 (in English).—The sensitivity of the method described in Part I (*Anal. Abstr.*, 1959, **6**, 1537) is increased, so that the present procedure can be applied in micro-determinations. The influence of several variables is investigated, and determinations of vitamin A in multivitamin - mineral tablets, cod-liver oil, animal liver, oleomargarine and serum are described. The extinction of the tungstophosphoric acid complex is read at 620 mμ, and not at 538 to 540 mμ, as previously. M. J. MAURICE

1246. **The determination of thiamine, riboflavine, pyridoxine and nicotinamide in multivitamin preparations. II. Application of the methods of analysis.** A. W. M. Indemans and H. E. J. Rademakers (Lab. Ned. Apothekers, Utrecht, Netherlands). *Pharm. Weekbl.*, 1960, **95** (13), 413–425.—The methods previously described (*Anal. Abstr.*, 1961, **8**, 783) are applied to multivitamin preparations, whereby interferences are eliminated by the method of the internal standard and by measuring against a suitable blank. *Procedures:* (*i*) *Determination of thiamine*—Dissolve an amount of powdered tablets containing $\not>$5 mg of riboflavine and $\not<$0·5 mg of pyridoxine by heating in about 75 ml of H_2O. Cool, add 250 mg of MnO_2, shake for 5 min. and dilute to 100 ml with H_2O. Filter this soln. (soln. *A*) and complete the determination by the thiochrome method. (*ii*) *Determination of riboflavine*—Dilute an aliquot of soln. *A* to a concn. of about 0·2 μg of riboflavine per ml. For the last dilution use a buffer soln. (pH 4·8). Measure the fluorescence of this soln. before and after addition of about 20 mg of $Na_2S_2O_4$. Also measure the fluorescence of the diluted soln. after addition of 0·2 μg of riboflavine (internal standard). (*iii*) *Determination of pyridoxine*—Dilute an aliquot of soln. *A* to a concn. of 5 to 15 μg of pyridoxine per ml. To 1 ml of this soln. add 1 ml of buffer (pH 9·5), 6 ml of isopropyl alcohol and 1 ml of 2,6-dibromo-*p*-benzoquinone-4-chlorimine (0·04% w/v in isopropyl alcohol). Exactly 5 min. after the addition of the last reagent measure the extinction at 650 mμ in a 1-cm cell against a blank prepared with 1 ml of 2·5% H_3BO_3 soln. instead of the sample soln. Compare with a standard. (*iv*) *Determination of nicotinamide*—Heat an aliquot of soln. *A* containing $\simeq$10 μg of nicotinamide per ml with NaBrO and phenol, diazotise and treat with ammonium sulphamate and *N*-1-naphthylethylenediamine hydrochloride. Measure the extinction at 500 mμ and compare with a standard. M. J. MAURICE

1247. **Physicochemical characterisation of vitamin-K homologues. I. Identification by a novel method of quantitative infra-red spectrophotometry.** H. Noll (Dept. of Microbiol., Sch. of Med., Univ. of Pittsburgh, Pa., U.S.A.). *J. Biol. Chem.*, 1960, **235** (8), 2207–2216.—A method is described for the rapid identification of vitamin-K analogues (K_1 homologues and K_2 homologues) by qual. and quant. i.r. spectrophotometry; the K_1 are phytyl, and the K_2 are polyisoprenoid, derivatives. Absorption bands near 1100 and 840 cm^{-1}, present in the K_2 but not in the K_1 spectra, permit qual. differentiation between the two homologous series. The length of the side-chain is determined within $\pm$0·2 isoprenoid unit by measurements of the relative intensities of certain bands. There is a linear relationship between the number of isoprenoid units in the side-chain and the ratio of the main alkyl stretching and bending bands to the extinctions of the characteristic naphthaquinone bands. These ratios are characteristic for each homologue. Independence of instrumental factors is obtained by a novel slit-width-calibration procedure with commercial DL-vitamin K_1 as the standard. The theoretical basis and limitations of the method and its general application to the quant. differentiation of qualitatively indistinguishable i.r. spectra are discussed. J. N. ASHLEY

See also Abstracts—**1056**, Pyrolysis and gas chromatography of olive oil. **1088**, EDTA in wine. **1195**, Determination of caffeine. **1267**, Hg in fruit and coffee. **1295**, Chromatography of lipids.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

1248. **Differential oxygen analyser.** C. U. Linderstrøm-Lang (Danish Atomic Energy Comm., Risø, Denmark). *Acta Chem. Scand.*, 1960, **14** (5), 1031–1036 (in English).—An apparatus is described for the determination of very small differences in oxygen content of air samples ($\simeq$200 ml). The oxygen is absorbed in alkaline pyrogallol soln. and the small amount of CO produced is determined and allowed for. Differences (0·1% of total pressure) can be determined with a standard deviation of 0·005% of the oxygen content. J. W. PRICE

1249. **Accurate determination of lower concentrations of carbon dioxide in air.** W. Deckert (Hygienisch. Inst. der Freien und Hansestadt, Hamburg, Germany). *Z. anal. Chem.*, 1960, **176** (3), 163–169 (in German).—A comparative study is made of the merits of available methods for the accurate determination of CO_2 in air, and it is shown that suitable absorption liquids are ethanolic KOH or NH_3 soln. at $-80°$. The titration of the carbonate formed is carried out with 0·1N-NaOH, with thymolphthalein as indicator, with a mean error of 0·5 to 2%. B. B. BAUMINGER

1250. **Use of membrane filters for the analysis of aerosols and industrial dust.** K. Spurný (Inst. Phys. Chem., Acad. Sci., Prague). *Chem. Listy*, 1960, **54** (9), 947–962.—A literature survey is given with descriptions of the apparatus and techniques used. (76 references.) J. Zýka

1251. **Analysis of micron-sized particles. Determination of sulphuric acid aerosol.** J. P. Lodge, jun., J. Ferguson and B. R. Havlik (Chem. Res. & Develop., Air Pollution Engng Res., Robert A. Taft Sanitary Engineering Center, U.S. Public Health Service, Cincinnati, Ohio). *Anal. Chem.*, 1960, **32** (9), 1206–1207.—A technique is described for the simultaneous detection of H^+ and SO_4^{2-}. The method is based on those of Jerhard and Johnstone (*Anal. Chem.*, 1955, **27**, 703) and Lodge and Fanzoi (*Ibid.*, 1954, **26**, 1829). K. A. Proctor

1252. **Estimation of total phosphate in water.** R. S. Robertson (Nalco Chem. Co., Chicago, Ill.). *J. Amer. Wat. Wks Ass.*, 1960, **52** (4), 483–491.—The normal test in which the complex condensed phosphate is converted into orthophosphate, followed by colorimetric methods, is discussed. The results obtained are often low, and complete degradation may require 1 to 4 hr. In the improved method described, the acid molybdate reagent (**I**) is prepared by adding 9·15 g of $(NH_4)_6Mo_7O_{24}.4H_2O$ (**II**) to 700 ml of water together with 1·5 g of $Bi(NO_3)_3.5H_2O$ dissolved in 191 ml of conc. HNO_3 and making up to 1 litre with water. The acid reagent is prepared similarly but omitting **II**. The water sample (3 ml) is added to each of two matched cells and 2 ml of **I** is added to one cell and 2 ml of acid reagent to the other. The contents of each cell are mixed and heated for $\simeq$5 min. till the max. yellow colour develops, the reaction being complete when the samples boil. They are then removed and 10 ml of water is added with 0·5 ml of reducing soln. (1-amino-2-naphthol-4-sulphonic acid, Na_2SO_3 and $Na_2S_2O_5$), and after 5 min. the colour is measured. There is no interference from tannins and lignins; interfering sulphide is removed by the addition of satd. bromine water; V, W, Sb and Sn can be tolerated at 10 mg per litre. The effects of various acids, and oxidising and reducing agents are reviewed, and the influence of Bi^{3+} and EDTA when added before and after the reduction is discussed. This method is intended for use with all soluble inorganic phosphates, but work with adenosine triphosphate showed that all the phosphate could be determined; by the normal method this is not possible without partially destroying the organic part of the molecule. (10 references.) S.C.I. Abstr.

1253. **A rapid method for determining fission products contained in waters using an ion exchanger.** Hiroyuki Tsubota and Yasushi Kitano (Nat. Inst. of Radiolog. Sci., Kurosuna-machi, Chiba). *Bull. Chem. Soc. Japan*, 1960, **33** (6), 765–769.—The separation of alkali, alkaline-earth and rare-earth elements contained in mixed fission products on Dowex 50-X8 resin (H^+ form) is described, with 0·2N-HCl, 0·5% oxalic acid soln. and formate buffer soln. (pH 3·2, 3·6, 3·8 and 4·2) as eluents. I. Jones

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See also Abstracts—**950**, Ba, Ca and Mg in water by h.f. titration. **991**, Phenoldisulphonic acid method for NO_3^-. **1330**, Liquid scintillation counting of water and effluents.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

1254. **Suppression of calcium interference in the flame-photometric determination of exchangeable sodium in soil.** C. H. Williams (Div. of Plant Ind., C.S.I.R.O., Canberra, Australia). *Anal. Chim. Acta*, 1960, **23** (2), 183–185 (in English).—Complete suppression of the interference in NH_4Cl extracts of soil containing up to 200 p.p.m. of Ca can be achieved by the addition of Al and P to give concn. >400 p.p.m. of each. G. S. Roberts

1255. **Estimation of molybdenum in soils and plants.** R. K. Chatterjee (Indian Agricultural Research Institute, New Delhi, India). *J. Sci. Ind. Res., India, C*, 1960, **19** (6), 154–155.—Molybdenum forms an orange-red complex in acid soln. with alkali thiocyanate and stannous chloride. This complex is extractable with various organic solvents, but only ethyl ether gives consistent results for quantitative determination. Iron, previously reported to interfere by intensifying the colour, has no effect when ethyl ether is the extractant. Silica interferes, but permissible limits are not given. For the determination of up to 10 p.p.m. of Mo in plant materials and soil, the sample is fused with Na_2CO_3 and the melt extracted with water. No procedural details are given beyond the use of a Lumetron 402E colorimeter with a M465 filter and a 2-cm cell for measuring the extinction. J. I. M. Jones

1256. **Factors affecting the estimation of phosphate esters in soil.** G. Anderson (Macaulay Inst. for Soil Res., Craigiebuckler, Aberdeen, Scotland). *J. Sci. Food Agric.*, 1960, **11** (9), 497–503.—Methods for the determination of organic phosphate in soil are critically reviewed, and experimental results obtained by three methods on a calcareous soil and an acid clay loam are discussed. The type of soil can influence the hydrolysis of some esters during extraction by standard procedures, and several types of esters are not recovered after acid pre-treatment. A modification of the procedure of Mehta *et al.* (*Proc. Soil Soc. Amer.*, 1954, **18**, 443) has been devised in which the soil is first extracted with 0·3N-NaOH, which recovers unchanged such esters as inositol hexaphosphate, glycerophosphate, glucose 1-phosphate and nucleic acids, which would otherwise be completely or partially hydrolysed during the acid-extraction stage. S. M. Marsh

1257. **Quantitative paper chromatography of chlorinated insecticides in soils.** J. P. San Antonio (Agric. Res. Service, U.S. Dept. Agric., Beltsville, Md.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 721–724.—A method previously studied (*J. Agric. Food Chem.*, 1959, **7**, 322) has been modified, whereby results affording an accuracy within about ±10% can be obtained. Some results for heptachlor, dieldrin, *pp'*-DDT, lindane and aldrin are recorded. A. A. Eldridge

1258. **Determination of humic acid in peat of balneological importance.** G. Sommer (Chem. Abteilung, Balneolog. Inst., Univ. München, Germany). *Z. anal. Chem.*, 1960, **176** (5), 347–355.—Problems of isolation, concentration and determination of humic acid in peat from thirteen different sources are described. E. G. Cummins

1259. **Determination of ammonia nitrogen in mixed commercial fertilisers by titration with sodium hypochlorite solution.** Z. Řezáč and M. Figarová (Res. Inst. for Inorg. Chem., Ústí nad Labem, Czechoslovakia). *Z. anal. Chem.*, 1960, **176** (2), 115–118 (in German).—To prevent pptn., Calgon S (condensed phosphate) is added, and the dead-stop method is preferred for the detection of the end-point. *Procedure*—The sample (10 g) is passed through a sieve (1 × 1), and 800 ml of water and 15 ml of conc. HCl are added. The mixture is shaken, made up to 1 litre and filtered. To 10 ml of the filtrate are added 65 ml of water and 5 ml of Calgon S soln. (5%) and the pH is adjusted with 20% NaOH soln. to 7 to 8; after addition of 5 g of $NaHCO_3$ and 0·5 g of KBr the soln. is titrated with 0·1N-NaOCl. This method gives results that agree favourably with those obtained by the distillation and potentiometric methods. The determination takes ≃15 min.
B. B. Bauminger

1260. **Determination of copper in fertilisers with 2,9-dimethyl-1,10-phenanthroline.** H. J. Webb and L. E. Nance (Clemson Agric. College, S.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 506–508.—Smith and McCurdy's method for the determination of Cu (*Brit. Abstr. C*, 1953, 105) can be successfully applied to fertilisers. The procedure is described. The coloured complex is stable for at least two months. A. A. Eldridge

1261. **Determination of total nitrogen and nitrogen distribution in fertiliser solutions.** J. D. Slater, J. S. Hill and D. C. Sesso (Grand River Chemical Div., Deere and Co., Pryor, Okla., U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 175–178.—Samples of fertiliser for determinations of N content were obtained by a modification of Greene's method. If only urea and NH_3 are present, NH_3 is determined by titrating to pH 4·0, with a pH meter. Urea is then determined by adding urease, and again titrating to pH 4·0 after 30 min. If nitrate is also present, distillable N is determined by adding NaOH, distilling into boric acid, and titrating to pH 4·7, with Sher's indicator (*Anal. Abstr.*, 1955, **2**, 2616). Nitrate N is then determined by adding Devarda's alloy, distilling into boric acid, and titrating again. Distillable N − urea N = ammonia N, and total N = distillable N + nitrate N. M. D. Anderson

1262. **Determination of total nitrogen in materials containing nitrate: reduction by chromous ion.** E. L. Nelson (Allied Chemical Corp. Res. Lab., Hopeville, Va., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 468–472.—Nitrate is reduced to NH_4^+ in a Kjeldahl digestion flask by addition of N-Cr^{II} soln., prepared (in an apparatus described and illustrated) by reduction of $CrCl_3$ with amalgamated zinc. Reduction of the nitrate is complete in 1 min. Average recoveries of nitrate from $NaNO_3$ or a synthetic nitrogen fertiliser soln. were 99·9%. The method is applicable to fertilisers containing nitrate and organic N with a high chloride content.
A. A. Eldridge

1263. **Phosphorus in fertilisers: direct determination of available phosphorus.** K. D. Jacob and W. M. Hoffman (Agric. Res. Service, U.S. Dept. Agric., Beltsville, Md.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 478–498.—Collaborative results are reported in detail and discussed. The methods employed were the direct photometric, volumetric ammonium molybdophosphate and gravimetric quinolinium molybdophosphate methods and the corresponding indirect methods, *i.e.*, determination of total P minus citrate-insol. P. The accuracy of the results was variable. Inorganic non-orthophosphate in fertiliser extracts can be converted into orthophosphate by mild acid digestion.
A. A. Eldridge

1264. **Micro-analysis of piperazine.** H. F. Beckman and L. Feldman (State Chemist Lab., Texas Agric. Exp. Sta., College Station, U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 227–228.—Piperazine used as an anthelmintic in feeds is determined by extracting with water or dilute acid, filtering through Super-Cel, adding 1,4-benzoquinone and ethanolic KOH, heating at 80° for 10 min., and measuring the orange-red product at 490 mμ. The recovery of piperazine added to feeds was 98 to 101%. Constituents of feeds, and other added medicaments, do not interfere.
M. D. Anderson

1265. **Determination of 3,5-dinitro-o-toluamide (Zoalene) in feed concentrates.** G. N. Smith (Dow Chemical Co., Midland, Mich., U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 224–226.—The method described for the determination of Zoalene in premix concentrates for adding to chick feeds is based on reaction with methylamine and dimethylformamide to give a purple complex, which is measured spectrophotometrically at 550 mμ. Methylamine may be replaced by propane-1,3-diamine, which gives a more stable complex and a more intense colour. The advantages of this method over those based on reduction, on reaction with acetone and alkali, and on reaction with KCN are discussed. The recovery of Zoalene averaged 97·2 ± 2·6%.
M. D. Anderson

1266. **Optimum conditions of hydrolysis for microbiological assay of amino-acids, methionine and cystine in poultry meat.** J. L. Fry and W. J. Stadelman (Purdue Univ., Lafayette, Ind., U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 243–244.—Poultry meat was hydrolysed with 3N-HCl by the sealed-tube method, or by heating under reflux, for 1 to 24 hr., and methionine and cystine were determined by acidimetric assay with *Leuconostoc mesenteroides*. Turbidimetric assay was found to be unsatisfactory. The sealed-tube method causes less destruction of the amino-acids. Maximum values were obtained for cystine after hydrolysis for 4 hr. in a sealed tube with 3N-HCl, and for methionine after hydrolysis for 8 hr. under the same conditions. A single hydrolysis for 6 hr. might give yields near the maximum for both amino-acids.
M. D. Anderson

1267. **Determination of mercury in plant material.** J. A. Pickard and J. T. Martin (Long Ashton Res. Station, Univ., Bristol, England). *J. Sci. Food Agric.*, 1960, **11** (7), 374–377.—A procedure that gives a high recovery of Hg from fruit and coffee beans is described. The sample is oxidised with HNO_3 - H_2SO_4 under carefully controlled conditions. The Hg is extracted at pH 5 with dithizone in the presence of $Na_2S_2O_5$ and EDTA as masking agent. The extinction is measured on the final soln. containing the Hg-dithizone complex at 490 mμ. Recoveries of up to 0·5 p.p.m. on a 50-g sample were satisfactory. B. Heining

1268. **Procedure for clean-up of plant extracts prior to analyses for DDT and related pesticides.** C. Anglin and W. P. McKinley (Food and Drug Directorate, Tunney's Pasture, Ottawa, Canada).

J. Agric. Food Chem., 1960, **8** (3), 186–189.—Waxes and pigments are removed from the sample before pesticide-residue analysis by using an acetone pptn. and Florisil column chromatography, respectively. *Procedure*—Evaporate the plant extract to about 10 ml on a water bath at 60° in a stream of oil-free air. Transfer the residue to a test-tube (20 cm × 2·4 cm) with benzene, and again evaporate until 3 ml of solvent is left. Add 20 ml of acetone, mix and cool at −70° (solid CO_2 and acetone) for 15 min. Then filter under suction, washing the residue with cold acetone (at −70°) (2 × 3 ml). Transfer the filtrate, with acetone, to a 100-ml beaker, add 5 g of anhyd. Na_2SO_4 and evaporate with stirring in a stream of air. Add this dry powdered residue to the top of a chromatographic column containing activated Florisil (8 in.) covered with 5 g of anhyd. Na_2SO_4. Rinse the beaker with benzene (4 × 5 ml) and add the rinsings, followed by 150 ml of benzene, to the column. Allow the column to run dry, and make up this wax- and pigment-free eluate to 200 ml. Aliquots can be used for qual. and quant. analysis of pesticides. The method has been used for all types of plant extracts, *e.g.*, from apples, cabbage, lettuce and oranges, and has been found suitable for clean-up before DDT analyses.

R. A. Hendey

1269. **Determination of *NN'*-diphenyl-*p*-phenylenediamine (DPPD) in fat and other biological materials.** P. Budowski, I. Ascarelli and A. Bondi (Agric. Res. Sta., Rehovot, Israel). *J. Sci. Food Agric.*, 1960, **11** (9), 503–509.—Some colour reactions of DPPD are described and discussed. A general procedure for determination of DPPD in biological materials is presented in which a light petroleum extract (50 ml ≡ ≯20 g of material) is purified by washing with phosphoric acid (3:1) and $SnCl_2$ soln.; the extract is evaporated almost to dryness *in vacuo* and a red colour is developed by treatment of the residue with the reagent (64 ml of conc. H_2SO_4, 2 ml of 69% HNO_3 and 36 ml of water) (5 ml). The extinction is measured at 540 mμ and compared with a calibration curve. Procedures for the extraction of DPPD from fat, chicken liver and other animal organs, egg yolk, mixed feed and blood serum are detailed. The sensitivity of the method is ≃1 μg. No losses were caused by the analytical procedure, heat treatment of the material or oxidation.

S. M. Marsh

1270. **Micro-determination of endrin.** E. J. Skerrett and E. A. Baker (Res. Sta., Long Ashton, Bristol, England). *Analyst*, 1960, **85**, 606–607.—A soln. of endrin in benzene is evaporated at 20° and 16 to 20 torr and the residue is heated at 60° with a specified acetic acid - H_2SO_4 mixture. Benzene (5 ml) and 10N-NaOH (2 ml) are added and the separated benzene layer is washed with water and dried with Na_2SO_4. Benzene is removed under reduced pressure at 60°, the residue is dissolved in ethanol (0·5 ml), and 0·1 ml of 2,4-dinitrophenylhydrazine reagent (Baker and Skerrett, *Ibid.*, 1960, **85**, 184) is added. After 30 min. the mixture, dissolved in 5 ml of benzene, is washed successively with conc. HCl (10 ml), N-NaOH (5 ml), conc. HCl 5 ml) and water (15 ml), and dried with Na_2SO_4, which is washed with benzene until the final vol. is 7·5 ml. Tetraethylammonium hydroxide reagent (prep. described) is added and the extinction is measured at 440 mμ after 1 min. (≯10 min.). The wt. (μg) of endrin present (B) is given by $A = 1{\cdot}40 \times 10^{-3}B$, A being the extinction. An extract of 100 blackcurrant flower buds treated with 0·5 g each of CaO and Draco G carbon was passed through a column of alumina covered with Na_2SO_4. The wt. of endrin found was 2·04 mg per bud, and the recovery of 200 μg added to an extract of unsprayed buds was 91%. Blank values were high.

A. O. Jones

1271. **Quantitative determination of Thiodan by gas chromatography.** G. Zweig and T. E. Archer (Pesticide Residue Res., Univ. of California, Davis, U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 190–192.—Thiodan has been determined by i.r. spectrography, or by hydrolysis followed by iodimetric or colorimetric determination of the liberated SO_2. Difficulties in these methods led to the investigation of the possibility of determining Thiodan by gas chromatography. No isomerisation or decomposition of Thiodan occurred on the column at 250°, as shown by i.r. spectrography, m.p., and elementary analysis. It was possible to separate the geometric isomers of Thiodan.

M. D. Anderson

1272. **Colorimetric method for determination of EPTC residues in crops and soil.** G. H. Batchelder and G. G. Patchett (Stauffer Chemical Co., Richmond, Calif., U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 214–216.—Residues of the herbicide EPTC (ethyl di-n-propylthiocarbamate) are separated from crops (*i*) by extracting with hexane, removing most of the solvent by rapid distillation through a 3-ball Snyder column, steam-distilling the EPTC and residual hexane, acidifying the distillate with HCl, and extracting the aq. phase with the hexane, or (*ii*) by macerating the sample with water, adding glacial acetic acid, steam-distilling, acidifying the distillate with HCl, and extracting the EPTC with iso-octane. The second method is used for some crops, and all soil samples. The EPTC is then hydrolysed by H_2SO_4, giving di-n-propylamine, which is extracted into benzene; the cupric dithiocarbamate complex is formed by reaction with CS_2 in the presence of NH_3 and Cu^{2+}, and measured spectrophotometrically at 440 mμ. Recovery of EPTC added to hexane extracts was 72 to 100%, and 0·02 p.p.m. can be determined. The method was successfully applied to numerous crops (except turnips, which contain interfering substances).

M. D. Anderson

1273. **Improved extraction procedure for the determination of EPTC [ethyl di-n-propylthiocarbamate] residues in potatoes.** W. H. Gutenmann and D. J. Lisk (Pesticide Residue Lab., Dept. of Entomology, N.Y. State Coll. of Agric., Cornell Univ., Ithaca, U.S.A.). *J. Agric. Food Chem.*, 1960, **8** (3), 216–217.—*Procedure*—Macerate about 12 potatoes in their own liquid in a 1-gall. blender. Weigh a 200-g portion into a 1-qt. blender and blend for 30 sec. with 200 ml of isopropyl alcohol. Add 300 ml of n-hexane and blend for 2 min. Filter the mixture through a large coarse-porosity sintered glass funnel, with suction. Rinse the residue with 25 ml of n-hexane, transfer the filtrate to a 500-ml separating-funnel and allow the layers to separate. Wash the lower aq. isopropyl alcohol layer with n-hexane (2 × 50 ml). Combine the hexane extracts and determine EPTC by the method of Batchelder and Patchett (*Anal. Abstr.*, 1961, **8**, 1272). The mean recovery of 0·1 p.p.m. of EPTC was 96·9% (6 determinations).

R. A. Hendey

1274. **The infra-red spectra of organic phosphate pesticides and their application to some problems in phosphate pesticide analysis.** D. F. McCaulley and

J. W. Cook (Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 710–717.—Peaks attributed to functional groups present in organic phosphate pesticides are discussed. Methyl cyanide is recommended as the most suitable extractant; the combined fractions are extracted with iso-octane, which is then re-extracted with methyl cyanide. After removal of the solvent the residue is subjected to short-path high-vacuum condensation under defined conditions, leading to reasonably satisfactory removal of the comparatively volatile pesticides and their metabolites. The condensate can be washed out with CS_2 and the spectra determined directly, but a "clean-up" procedure with a Florisil column may be desirable. These observations may lead to a general qualitative or semi-quantitative method of analysis of phosphate pesticides and their metabolites.

A. A. ELDRIDGE

1275. **Residues of *OO*-dimethyl *S*-(methylcarbamoylmethyl) phosphorothiolothionate (dimethoate) in sprayed crops.** E. D. Chilwell and P. T. Beecham (Fisons Pest Control Ltd., Saffron Walden, Essex, England). *J. Sci. Food Agric.*, 1960, **11** (7), 400–407.—The macerated sample is acidified to pH 4 with 10% acetic acid and filtered; maceration and filtration are repeated twice. The filtrates are combined, centrifuged and made up to 1 litre with water. A 200-ml aliquot is neutralised with 2N-NaOH and extracted twice with an equal vol. of chloroform. The combined extracts are filtered and the chloroform distilled down to about 10 ml in a special micro-distillation flask. After removing the remainder of the solvent at a low temp. the pressure is reduced to 1 torr and the contents of the flask are kept at 200° for 30 min. The dimethoate is condensed on a cold finger which is washed into a 50-ml Kjeldahl flask with 5 ml of water; 2 ml of 72% $HClO_4$ is added and the flask is heated to remove chloroform. Then 5 drops of HNO_3 are added and evaporated to fuming; after cooling, a little water is added and the evaporation is repeated. The phosphorus is determined colorimetrically by any suitable method and calculated as dimethoate. The limit of sensitivity of the method is about 5 μg of dimethoate.

B. HEINING

1276. **Determination of 2,2-dichlorovinyl dimethyl phosphate (DDVP).** M. Geiger and R. Fürer (Plant Protection Res. Lab., CIBA A.-G., Basel, Switzerland). *Z. anal. Chem.*, 1960, **174** (6), 401–407.—Three methods are described. For the macro-determination, DDVP is hydrolysed to dichloroacetaldehyde (**I**) and dimethylphosphoric acid with 0·4N-NaOH in the presence of 0·02N-iodine; the amount of iodine consumed in oxidising **I** to dichloroacetic acid is a measure of the DDVP and is determined with $Na_2S_2O_3$. Alternatively, DDVP is hydrolysed with a measured excess of alkali at 0° to 1° (no iodine present) and the excess is determined by back-titration with acid; interference by **I** is avoided by careful adjustment of conditions. The accuracy is within $\pm 0{\cdot}5\%$. For the micro-determination, an aq. soln. of DDVP is treated with alkaline resorcinol soln. and the colour is measured after 4 hr. at 20°, or 45 min. at 40°, at 480 mμ. This method is sensitive down to 5 μg, and is accurate to within $\pm 5\%$; **I** interferes.

J. P. STERN

1277. **Paper chromatography of ferbam, maneb, nabam, thiram, zineb and ziram.** W. P. McKinley and S. A. Magarvey (Food and Drug Lab., Dept. of Nat. Health and Welfare, Ottawa, Canada). *J. Ass. Off. Agric. Chem.*, 1960, **43** (3), 717–720.—A chromatographic procedure for the resolution of these pesticides into two groups is described; formamide in acetone is used as the stationary phase and $CHCl_3$ as the mobile phase, when ferbam, thiram and ziram move to the solvent front. Feigl's sodium azide - iodine reaction catalysed by thioketones is used for detection.

A. A. ELDRIDGE

See also Abstracts—**1185**, Determination of Trithion and Phosdrin. **1237**, 1-Chloro-2-nitrobenzene in pineapple. **1239**, DDT in flour. **1240**, Heptachlor epoxide in fats and milk. **1241**, Pesticide residues in foods.

5.—GENERAL TECHNIQUE AND APPARATUS

General

1278. **Analysis of gas evolution from a titanium hydride gas generator.** L. Levine and D. Lichtman (Sperry Gyroscope Co., Gt. Neck, Long Is., N. York). *Rev. Sci. Instrum.*, 1960, **31** (7), 731–733.—Mass-spectrometric analyses have been made to determine the purity of the hydrogen evolved from a titanium hydride generator before, during and after bake-out, and during normal running conditions.

G. SKIRROW

1279. **Weighing technique for substances that are very volatile and unstable in air.** J. Jeník and J. Churáček (Inst. Chem. Technol., Pardubice, Czechoslovakia). *Chem. Listy*, 1960, **54** (9), 966–967.—For the micro-determination of Si in organosilicon compounds a new technique was developed for weighing alkyl- and aryl-halogenosilanes. The samples are stored in 10-ml penicillin flasks in an atmosphere of N, and the sampling is carried out with a 1-ml syringe into capsules (15 mm × 1 mm) made from polyethylene tubes.

J. ZÝKA

1280. **Analytical apparatus [for the quantitative determination of elements in organic compounds].** Loughborough Glass Co., Ltd. [Inventor: I. K. H. Otter]. Brit. Pat. 850,874; date appl. 26.3.58.—The apparatus comprises a quartz bulb provided with an externally extending combustion tube opening only into the bulb, and an absorption vessel enabling gases from the bulb to be passed through absorbent liquid contained in the absorption vessel. The absorption vessel comprises a tube and a reservoir, the tube leading from the bulb of the analytical apparatus into the reservoir, which has a filling device capable of being sealed by a stopper.

J. M. JACOBS

1281. **A new pH meter cell assembly.** P. Bamford (I.C.I. Ltd., Akers Research Lab., Welwyn, England). *Lab. Practice*, 1960, **9** (8), 597–598.—The cell (illustrated) is a glass cylinder fitted with a tap and filling side-funnel. A rubber bung carries the glass and calomel electrodes, a thermometer and four glass capillaries supplying the necessary reagents. This assembly is mounted on a vertical slotted bar attached eccentrically to a wheel driven by a small motor. The pH meter is standardised, then the test soln. (6 to 20 ml) is poured in and the

pH read. The appropriate reagent is added drop-wise, with the shaker running, until the required pH is reached. The cell enables the routine adjustment of large numbers of small aq. samples to be made quickly and accurately. R. A. HENDEY

1282. **Precise automatic inexpensive burette reader.** J. Farquharson (Oak Ridge Gaseous Diff. Plant, Union Carbide Nuclear Co., Tenn., U.S.A.). *Rev. Sci. Instrum.*, 1960, **31** (7), 723–725.—The instrument is essentially a meniscus-seeking device consisting of a photocell and light source which traverses the burette tube by means of a cable and drum-elevating mechanism. Movement is controlled by a servo motor which also operates a digital register. A precision of ± 0.02 ml on a single determination and a bias of 0·1% is claimed. G. SKIRROW

1283. **Electromagnetic burette valve with two rates of delivery.** J. T. Stock and M. A. Fill (Norwood Tech. Coll., London, S.E.27). *Analyst*, 1960, **85**, 609–610.—In the valve described, a single compression-type control is used with two adjustable degrees of opening according to the energising of one or both of two electromagnets. The low rate of flow can be adjusted to a rate of one drop in several seconds. A. O. JONES

1284. **Use of automatic apparatus in titrations with potassium permanganate.** G. Halfter and W. Kuttler (Geigy A.-G., Grenzach/Baden, Germany). *Z. anal. Chem.*, 1960, **176** (2), 111–115 (in German). —The automatic titration of 0·01N-$KMnO_4$ and the determination of Mn are discussed. The apparatus can be used for the study of reaction mechanisms. B. B. BAUMINGER

1285. **Two-stage micro-evaporator.** E. G. C. Clarke and A. E. Hawkins (Dept. of Physiol., Royal Veterinary Coll., London). *J. Pharm. Pharmacol.*, 1960, **12** (8), 509–511.—Two devices are described. The first serves to reduce a volume of 1 ml to a volume of 0·05 ml; it works best with $CHCl_3$ or methanol as solvent, although it may be used with less volatile solvents such as ethanol or H_2O. It is especially useful for the concentration of a solution containing material eluted from a paper chromatogram. The second device serves to reduce a volume of 0·05 ml to a few microlitres. A. R. ROGERS

1286. **Temperature regulator for close temperature control.** E. N. Hertzberg (Min. of Defence Scientific Dept., Hakiya, Tel Aviv, Israel). *J. Sci. Instrum.*, 1960, **37** (8), 285–289.—Circuit diagrams and performance details are given for an electronic oven-temp. regulator having a re-settability better than 1° in the temp. range 150° to 550° and peak-to-peak temp. fluctuations $<0.015°$. G. SKIRROW

1287. **Characterisation of reference liquids used for the determination of critical solution temperatures.** R. Fischer and T. Kartnig (Inst. f. Pharmakog., Univ. Graz, Austria). *Pharm. Zentralh.*, 1959, **98** (7), 366–370.—The purity of H_2O, benzene, nitrobenzene, aniline and diethyl sulphate is established by determination of b.p. and refractive index. Butane-1,3-diol, heptane, liquid paraffin, olive oil and other reference liquids are best characterised by measurement of the critical solution temp. of mixtures. Constants of over 50 reference liquids are tabulated. (*Cf.* Fischer and Resch, *Anal. Abstr.*, 1955, **2**, 3183.) A. R. ROGERS

1288. **Evaluation of particle-size distributions obtained from electrolytic resistivity changes.** R. R. Irani (Res. Dept., Inorg. Chem. Div., Monsanto Chemical Co., St. Louis, Mo., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1162–1164.—A critical study has been made of particle-size distributions by comparing results obtained from measurement of changes in electrolytic resistivity with those obtained by sedimentation and electronic microscopic sizing and counting. The first method gave results significantly different from the others. For some powders a calibration procedure is proposed. K. A. PROCTOR

See also Abstracts—**1038**, Preparation of silver crucibles. **1062**, Unit for oxygen flask combustion method.

Chromatography, ion exchange, electrophoresis

1289. **Automatic device for changing solutions in column chromatography.** J. H. Kreisher and J. H. McClendon (Dept. of Agric. Biochem. and Food Technol., Univ. of Delaware, Newark, U.S.A.). *Anal. Chem.*, 1960, **32** (7), 890–891.—Solenoid-actuated glass valves are used in the device described which enables eluent buffers going into ion-exchange columns to be changed automatically on a reproducible schedule. Up to 20 steps may be made within one column run and gradients may be used together with linear steps. The system has several possible uses in routine amino-acid separations and in other procedures in which soln. changes are required. K. A. PROCTOR

1290. **Gradient elution chromatography.** O. Mikeš (Inst. Org. Chem. and Biochem., Acad. Sci., Prague). *Chem. Listy*, 1960, **54** (6), 576–592.—Principles, techniques, theory and possible applications of this chromatographic method are reviewed. (51 references.) J. ZÝKA

1291. **Application of a nomogram to gradient elution chromatography.** H. R. Warner and W. E. M. Lands (Univ. Michigan, Ann Arbor, U.S.A.). *J. Lipid Res.*, 1960, **1** (3), 248–250.—A nomogram is described for the determination of the composition of the solvent leaving the mixing chamber in constant-volume gradient elution chromatography with a two-component solvent system. From the results for gradient elution the compositions of suitable solvent mixtures for stepwise elution can be calculated. The separation of ovolecithin from lysolecithin on silicic acid with a $CHCl_3$ - methanol solvent system is described. R. E. E.

1292. **Further applications of electrostatic discharge current (Es. d.). VII. Moisture gauge for chromatograms.** G. G. Blake (Sydney Univ., Australia). *Anal. Chim. Acta*, 1960, **23** (1), 10–12 (in English).—The apparatus described is an aid to the control of the moisture content of paper chromatograms for purposes of zone location by conductivity measurements. A strip of filter-paper impregnated with $CoCl_2$, or other hygroscopic salt, is mounted between two terminals in series with a galvanometer. A condenser is discharged through the circuit and the max. meter reading provides a measure of the humidity of the atmosphere in which the strip is placed. The device can be used in the humidiser previously described (*cf. Anal. Abstr.*, 1960, **7**, 4054). H. N. S.

1293. **Chromatography on paper impregnated with zirconium phosphate.** G. Alberti and G. Grassini (Ist. di Chim. Gen. ed Inorg., Lab. Chim. della Div. Geomineraria del C.N.R.N., Roma, Italy). *J. Chromatography*, 1960, **4** (1), 83–85 (in English).—Strips (6 cm × 40 cm) of Whatman No. 1 filter-paper impregnated with zirconium phosphate (details given) are used for the separation of 26 cations. The strips are developed for 4 to 8 hr. by the ascending technique with various concn. of HCl, NH_4Cl - HCl, NaCl - HCl and KCl - HCl. R_F values for the cations in 0·1N-, 0·5N- and N-HCl are listed. E. C. BUTTERWORTH

1294. **Chromatography on paper impregnated with inorganic ion-exchangers.** J. M. Peixoto Cabral [Inst. de Alta Cultura-C.E.E.N., Centro de Quim. (I.S.T.), Lisbon, Portugal]. *J. Chromatography*, 1960, **4** (1), 86–87 (in English).—Chromatographic separation of ^{45}Ca, ^{89}Sr and ^{140}Ba in 0·5N-HCl is achieved with Whatman No. 3MM paper strips (3 cm × 25 cm) impregnated with zirconium molybdate and developed for 20 cm with 0·9N-NH_4Cl by the ascending technique. The separation of ^{137}Cs from the radionuclides of Ca, Ba and Sr is carried out similarly with zirconium phosphate paper strips developed with 0·1N to 0·5N-HCl for 15 cm. E. C. BUTTERWORTH

1295. **Chromatographic separation of lipids on paper made from glass fibres.** M. Černiková (Trýb's Lab. für Biochemie der Haut, Masaryk-Univ., Brno, Czechoslovakia). *Fette, Seif., Anstrichmitt.*, 1960, **62** (7), 587–589.—The effects of solvents, impregnation, pH, quantity of substance applied, speed of flow and temp. on the chromatographic analysis of lipids were studied. It was found that most of these variable factors had little effect on the separation of lipids. It is, however, important to carry out the instructions given for the impregnation of the paper (140 g of silica gel in 1 litre of water is used), and to make sure that the chamber is saturated completely. I. DICKINSON

1296. **Improved glass atomiser for paper chromatography.** S. Bose, S. Mukherjee and J. N. Sharma (Nat. Sugar Inst., Kanpur, India). *Sci. & Cult.*, 1960, **26** (1), 44.—An all-glass apparatus is described and illustrated in which the spraying unit and the liquid container are combined. It can deliver a good volume of fine spray, requires only a low pressure for operation, and is convenient for spraying large chromatograms. R. E. E.

1297. **Scintillation counting of paper chromatograms.** R. B. Loftfield and E. A. Eigner (J. C. Warren Lab., Huntington Mem. Hospital, Harvard Univ., Cambridge, Mass., U.S.A.). *Biochem. Biophys. Res. Commun.*, 1960, **3** (1), 72–76.—A procedure is described for the measurement of the ^{14}C activity of compounds such as valine and valine hydroxamate. The chromatograms are prepared on thin Amberlite SA cation-exchange paper, on which valine moves with the solvent front and valine hydroxamate remains at the origin (Loftfield and Eigner, *J. Amer. Chem. Soc.*, 1959, **81**, 4753). After development with 0·5M-sodium phosphate buffer, the paper is dried, and 2-in. sections are cut out at the origin and at the solvent front, rolled into cylinders and inserted in test-tubes filled with the scintillation solvent. The activity is then measured in a Packard Tri-Carb counter at Tap 6, 10 to 50 V. This is claimed to be ten times as efficient as an end-window Geiger - Müller counter, and determinations are accurate to within 3% over the range 100 to 80,000 counts per min. R. E. E.

1298. **Expedient for increasing capacity of paper-chromatography tanks.** R. Nakamura, S. Kushinsky and J. Wu (School of Med., Univ. of S. Calif., Los Angeles). *Chemist Analyst*, 1960, **49** (2), 54.—The capacity of a tank, for use with the descending technique, in which a sheet of paper is supported between two glass plates extending over the edge of the solvent trough, can be doubled by supporting two papers between three plates, of which the upper two extend beyond the lowest. The difference in mobility can be compensated by using standards on each paper. G. BURGER

1299. **Gas chromatography for mine-gas analysis.** A. R. Baker and F. J. Hartwell (S.M.R.E., Ministry of Power, Sheffield). *S.M.R.E. Res. Rep.*, 1960, No. 189, 31 pp.—The general principles of gas chromatography are outlined and some applications to the analysis of mine gases are described. Details are given of three instruments built at S.M.R.E. for the estimation of many of the gases found in mine air. One is designed for the estimation of hydrocarbons other than methane, and contains three chromatographic columns operated in series. The second is for some of the more common gases, and the third is used to obtain reliable estimations of carbon monoxide. It is possible to detect 0·01% of most of the hydrocarbons and 0·001% of hydrogen and carbon monoxide. A complete analysis takes about 1 hr. The defects and errors in the present apparatus are discussed and improvements are suggested. FUEL ABSTR.

1300. **Reaction gas chromatography.** F. Drawert, R. Felgenhauer and G. Kupfer (Bundesforschungsanstalt für Rebenzüchtung, Geilweilerhof, Siebeldingen über Landau/Pfalz, Germany). *Angew. Chem.*, 1960, **72** (16), 555–559.—The apparatus used is described in detail. Gas chromatography is preceded by reactions designed to achieve conversion of the test substances into compounds readily separable in the chromatographic column, to which the reaction vessel is directly attached. Aq. alcohol mixtures are analysed by conversion of the alcohols into nitrous acid esters (*cf.* Drawert and Kupfer, *Anal. Abstr.*, 1960, **7**, 4326), or by dehydration to olefins, or by catalytic hydrogenation to paraffins. Blood alcohol can be accurately determined by the olefin method. E. G. CUMMINS

1301. **Gas-chromatographic analysis of volatile components in the presence of excess of non-volatiles.** E. W. Cieplinski and L. S. Ettre (Perkin-Elmer Corp., Norwalk, Conn., U.S.A.). *J. Chromatography*, 1960, **4** (2), 169–171 (in English).—The device described operates by condensing trace components of interest in a small trap-column connected to the outside gas-sampling valve of a gas-chromatographic column. The column has a rubber septum on top through which the sample is injected, and is filled with an adsorbent that has a longer retention time for the solvent than the dissolved substances to be analysed. The non-volatile solvent is adsorbed on the packing and the volatile components can be transferred without loss on to the chromatographic column. The column is then back-flushed with N or air. The method has been successfully applied to the analysis of dissolved gases or light organic solvents in water. S. M. MARSH

1302. **Automatic gas-sampling device for gas chromatography.** D. W. Hill and J. R. Hook (Royal Coll. of Surgeons, Lincoln's Inn Fields, London, England). *J. Sci. Instrum.*, 1960, **37** (7), 253-255.—The sample gas passes through a sampling loop milled on the inner surface of one of two closely-fitting stainless-steel discs. Rotation of one of the discs through 20° causes the carrier gas to sweep the sample on to the column. Operation of the mechanism is automatic and samples can be taken and analysed at regular time intervals.
G. Skirrow

1303. **Sampling valve for use in gas-chromatographic analysis of the products of gaseous reactions.** G. L. Pratt and J. H. Purnell (Dept. of Phys. Chem., Univ. of Cambridge, England). *Anal. Chem.*, 1960, **32** (9), 1213.—The valve described enables vapour samples to be removed directly from a vacuum system for injection into a gas-chromatographic analysis unit operating at high column-inlet pressures. K. A. Proctor

1304. **Stationary liquid phases for use in gas-liquid chromatography suitable for the separation of the components of essential oils.** R. A. Bernhard (Dept. Food Sci. and Technol., Univ. California, Davis, U.S.A.). *Food Res.*, 1960, **25** (4), 531-537.—Forty liquids were examined, and the results are tabulated. The liquid (25%) was supported on Sil-O-Cel C-22 diatomaceous earth firebrick (30 to 60 mesh) in a stainless-steel column equipped with a hot-wire 4-channel katharometer. For the separation of the components of cold-pressed lemon oil the best liquids were Craig polyester adipate, LAC-2-R446 and LAC-4-R777. (*Cf. Anal. Abstr.*, 1961, **8**, 201.) R. E. E.

1305. **Effect of the surface area on the separation in gas-liquid partition chromatography.** L. S. Ettre (Perkin-Elmer Corp., Norwalk, Conn., U.S.A.). *J. Chromatography*, 1960, **4** (2), 166-169 (in English). —The specific surface area and separation effect of five support materials, *viz*, firebrick, Chromosorb, Chromosorb W (Type 6), Celite 545 and Teflon, were determined and compared for a mixture of hydrocarbons and one of alcohols. It is concluded that the specific surface area has little effect on separation over a certain limit ($\simeq$1 sq. metre per g), when polarity has the greater influence. In general, Chromosorb, Celite and Chromosorb W give similar separation effects, firebrick has no advantages, and Teflon is preferable only with samples of extremely high polarity. S. M. Marsh

1306. **Plate height in programmed-temperature gas chromatography.** H. W. Habgood and W. E. Harris (Univ. Alberta, Edmonton, Canada). *Anal. Chem.*, 1960, **32** (9), 1206.—The definition of plate height in programmed-temp. gas chromatography is derived from the fundamental ideas of isothermal chromatography. K. A. Proctor

1307. **Plate height in programmed-temperature gas chromatography.** G. H. Stewart (Dept. Chem., Gonzaga Univ., Spokane, Wash., U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1205.—The meaning and usefulness of the plate-height concept in programmed-temp. gas chromatography is discussed.
K. A. Proctor

1308. **Removal and identification of organic compounds by chemical reaction, in [gas] chromatographic analysis.** R. Bassette and C. H. Whitnah (Kansas State Univ., Manhattan, U.S.A.). *Anal. Chem.*, 1960, **32** (9), 1098-1100.—Gas chromatograms were run before and after addition of $NaHSO_3$ and/or $HgCl_2$ to the sample soln. Comparison of the chromatograms showed that peaks due to all the carbonyl compounds, with the exception of ethyl methyl ketone, were eliminated by the addition of the bisulphite; the methyl sulphide peak was eliminated by the reaction with $HgCl_2$. The compounds studied were—acetaldehyde, propionaldehyde, dimethyl sulphide, acetone, isopropyl alcohol, n-butyraldehyde and ethyl methyl ketone, singly and in admixture. The substances were extracted into CCl_4 before the gas-chromatographic analysis. G. P. Cook

1309. **Electro-mechanical integrator for gas chromatography.** H. E. Dubský and J. Sokolíček (Lab. Gas Analysis, Acad. Sci., Brno, Czechoslovakia). *Chem. Listy*, 1960, **54** (7), 724-729.—The description of the apparatus and a circuit diagram are given.
J. Zýka

1310. **Mounting tungsten-filament katharometers for gas-chromatography columns.** A. Kreyenbuhl (Centre d'Études et Recherches des Charbonnages de France, Verneuil-en-Halatte). *J. Chromatography*, 1960, **4** (2), 130-137 (in French).—Detailed descriptions with diagrams are given of the construction of tungsten-filament katharometers for glass tubes of normal size and for larger all-metal tubes. S. M. Marsh

1311. **Chromatography of organic compounds. VI. A photo-detector for gas chromatography.** J. Franc (Forschungsinst. f. org. Synth., Pardubice-Rybitví, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2225-2227 (in German).—A photomultiplier is used instead of a photocell. The multiplier is 10^3 to 10^4 times as sensitive as a photocell and consequently permits the use of a less sensitive recorder and smaller samples. A special burner is described which avoids the mixing of the carrier gas (N) and fuel gas (H) below the jet of the burner. The burner is enclosed in a case to prevent the entry of interfering light and dust particles. The case also holds the reflector and an u.v. absorption filter. The N was used at a flow rate of 1 litre per hr., and the H at 2·5 to 3 litres per hr. In spite of the absence of optics between the photomultiplier and the burner, the sensitivity is 100 V per mg per ml for aromatic compounds and $\simeq$10 V per mg per ml for aliphatic compounds.
B. Heining

1312. **Simple apparatus for electrophoresis for various purposes.** M. Šťastný and E. Nováková (Lab. Metabolism of Proteins, Med. Fac., Charles' Univ., Prague). *Chem. Listy*, 1960, **54** (8), 850-851. —A new arrangement is described, suitable for the rapid separation of the protein fractions (mainly blood lipoproteins) obtained in the routine analysis of the protein spectrum of serum. Two Novodur or glass vessels (10 cm × 7·5 cm × 6·5 cm) are used as electrode troughs, into which nylon-sponge blocks (16 cm × 10 cm × 7·0 cm) are inserted so as to fill them tightly. The electrode system consists of two platinum wires inserted into the sponges saturated with buffer soln. The electrophoresis is carried out in agar gel on a microscope slide, or on Whatman No. 1 paper, laid with the ends on the sponges.
J. Zýka

1313. **Device for continuous flow of small amounts of samples in continuous paper electrophoresis.** C. Loštický (Inst. Haematology and Blood Transfusion, Prague). *Chem. Listy*, 1960, **54** (7), 729-730.

—The device enables samples to be applied continuously in small portions to the paper. A detailed description with figures is given. J. ZÝKA

1314. **Distortion-free zone electrophoresis in starch gel.** B. Paletta (Med. Chem. Inst. u. Pregl-Lab. d. Univ., Graz, Austria). *Clin. Chim. Acta*, 1960, **5** (4), 490–496 (in German).—An apparatus is described that provides uniform cooling and a homogeneous electric field. The sharpness of separation of protein fractions is illustrated. H. F. W. KIRKPATRICK

1315. **Transparent starch gels: preparation, optical properties and application to haemoglobin determination.** W. B. Gratzer and G. H. Beaven (Med. Res. Council Lab., Mill Hill, London, England). *Clin. Chim. Acta*, 1960, **5** (4), 577–582.—After completion of electrophoresis the gel is placed in undiluted glycerol and left for some hours, preferably overnight, in the cold. The gel is then almost completely transparent, somewhat shrunken and reduced in thickness, and extremely tough. Some applications are given, particularly with reference to the determination of haemoglobin A_2. Modified staining procedures for protein fractions in the cleared starch gel are discussed. H. F. W. KIRKPATRICK

1316. **Investigation of the conditions of separating substances by high-voltage electrophoresis on paper.** Z. Prusík and B. Keil (Inst. of Chem., Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (8), 2049–2058 (in English).—The construction of a cooled high-voltage paper-electrophoresis apparatus which operates at potential gradients up to 120 V per cm is described. With this apparatus, it is possible to obtain a reproducible degree of moisture in the paper and a constant temp. and to measure the potential gradient in the paper, and thus to measure the mobility of compounds directly. Details are given of the procedure used, and of studies of the electrophoretic mobility of various amino-acids, peptides and dinitrophenyl derivatives as a function of pH and the ionic strength of the medium. B. HEINING

1317. **Measurement of proteins separated by electrophoresis in starch gel.** H. J. Rubinstein, J. A. Owen and W. Larsen (Dept. of Biochem., Univ. Melbourne, Australia). *Clin. Chim. Acta*, 1960, **5** (4), 583–590.—Methods are described for rendering the gel transparent, for scanning the stained transparent gels with a specially constructed instrument, and for elution of the dye. Results are given. H. F. W. KIRKPATRICK

1318. **Staining of paper-electrophoresis strips. Comparison of azocarmine and Ponceau S staining techniques.** O. Meulemans (Dept. of Pediatrics, State Univ., Utrecht, The Netherlands). *Clin. Chim. Acta*, 1960, **5** (4), 615–616.—Staining with Ponceau S. (C.I. Acid Red 9) (0·15% in 3% trichloroacetic acid) and washing with 10% acetic acid followed by a single wash with H_2O gave results needing no correction factor, as albumin and the globulins apparently take up the same proportion of this dye. Measurement of the colour is made at 550 mμ after elution from the paper with 0·1N-NaOH. H. F. W. KIRKPATRICK

1319. **Direct photometric evaluation of electropherograms on dry films.** J. Barrollier (Schering A.-G., Berlin, Germany). *J. Chromatography*, 1960, **4** (2), 99–107 (in German).—Three methods for preparing transparent electropherogram films are described. (*i*) *Electrophoresis in a transparent medium*—Details of procedures with films of agar gel supported on a p.v.c. - chlorinated rubber foil, and of collodion gel supported on celluloid or cellulose acetate are given. The latter medium gives a very transparent film (84 to 88% at 400 to 600 mμ). (*ii*) *Electrophoresis in a medium that is subsequently converted to a transparent film*—Electrophoresis is carried out on cellulose acetate films with a technique similar to that used for paper electrophoresis. The electropherograms are then developed and sprayed with glacial acetic acid, when they form a transparent film. Completely transparent films are obtained by subsequently spraying with phenol - water - glacial acetic acid (8:2:1). Completely acetylated paper can also be used and treated with phenol - anisole - propane-1,2-diol, but the films obtained are less transparent. (*iii*) *Copying process*—A developed paper electropherogram is placed in contact with a specially prepared gelatin film between two electrodes in a buffered medium, pH 4·0. A potential of 10 to 15 V is applied, initially for 10 min. and then for periods of 5 min., until a satisfactory copy is obtained. The film is fixed for 20 to 30 min. with 95% methanol - glycerol (or propane-1,2-diol) - 40% formalin - glacial acetic acid (84:5:10:1). More than one copy can be made from a single electropherogram. S. M. MARSH

1320. **Modified tank for paper electrophoresis.** D. A. Osborn (Dept. of Pathology, Inst. of Laryngology and Otology, Univ. of London, England). *Lab. Practice*, 1960, **9** (8), 595–596.—An apparatus is described which provides for the soaking of 6 strips (2·5 cm × 20 cm) *in situ*, in a pre-saturated atmosphere. Diagrams and full details of the Perspex tank and electrode system are given. Advantages over conventional tanks are that the strips are always in a saturated atmosphere and there is no concentration gradient of the electrolyte across the strip. Both 3MM paper and cellulose acetate membrane can be used with equal success in the tank. R. A. HENDEY

1321. **Circular device for the calculation of the results of electrophoresis of blood serum on paper carrier.** O. Quadrát (Centr. Lab. Hospital Bulovka, Prague). *Chem. Listy*, 1960, **54** (8), 851–852.—The device described and illustrated consists of a fixed circular scale and two rotating circular scales, all logarithmic. The computation is based on the extinctions of the protein fractions. J. ZÝKA

See also Abstract—**896**, Automatic analysis by gas chromatography.

Optical

1322. **Improvements in spectrometers.** R. F. Jarrell and W. G. Fastie. Brit. Pat. 849,347; date appl. 27.5.57.—The apparatus comprises a first (prism) spectrometer that has means for dispersing rays from a source of radiant energy to provide a spectrum focused at the entrance slit of a second spectrometer, which includes means (*e.g.*, a spherical concave mirror) for parallelising the rays entering through the slit, means (*e.g.*, a plane reflecting diffraction grating) for dispersing these rays to form a second spectrum and means for recording

this second spectrum (*e.g.*, a series of photomultiplier tubes). These tubes and the entrance slit are located in the same vertical plane, the dispersing equipment of the second spectrometer being located between this plane and the parallelising equipment.
J. M. JACOBS

1323. **New spectrograph with plane diffraction grating.** P. Kröplin. *Chim. Anal.*, 1960, **42** (9), 448–453.—The basic principles are discussed and compared with those of concave gratings and prisms, and details are given of the operation of a recent German instrument.
J. H. WATON

1324. **The effect of spectroscopic buffers on the volatilisation rates of some elements.** E. van Rooyen (Div. of Chem. Services, Pretoria). *S. Afr. J. Agric. Sci.*, 1960, **3** (2), 163–169.—The buffers used in this investigation were $CaCO_3$, Al_2O_3 and Li_2CO_3, and their effects on Pb, Zn, Bi, Co, Sn, Al, Ge and Mo were studied. The sample for arcing contained two parts of standard sample (500 p.p.m. each of Co, Pb, Bi, Al, Sn and Ge, 100 p.p.m. of Mo and 2000 p.p.m. of Zn) and one part of the selected buffer. The moving-plate technique was used; excitation, recording conditions and the apparatus are described, and the wavelengths for the various elements are given. From the volatilisation curves obtained it was found that the elements can be classified as (*i*) highly volatile (Zn and Pb), (*ii*) moderately volatile (Sn, Co, Al, Ge and Bi), and (*iii*) poorly volatile (Mo). It is concluded that $CaCO_3$ is an efficient buffer for the determination of poorly volatile elements and that Li_2CO_3 is unsuitable for the determination of Co, Zn, Pb and Mo. Careful selection of the buffer eliminates the necessity for more than one internal standard.
I. DICKINSON

1325. **Stray-light error in a spectrophotometer.** H. Hoch, M. Turner and R. C. Williams (Med. Coll. of Virginia, Richmond, U.S.A.). *Clin. Chem.*, 1960, **6** (4), 345–351.—A correction formula is derived and applied to relate apparent absorption and concn.
H. F. W. KIRKPATRICK

1326. **Test-tube adapter for use in optical density measurements.** J. B. Dawson (Dept. of Med. Phys., Gen. Infirmary, Leeds, England). *J. Sci. Instrum.*, 1960, **37** (8), 304.—The test-tube fits into a parallel-sided adapter, the space between the two being filled with toluene. The close similarity of the refractive indices of glass and toluene results in improvement of the reproducibility of extinction measurements made with the adapter described.
G. SKIRROW

1327. **Small-volume long-path infra-red cell for liquids.** D. S. Erley, B. H. Blake and W. J. Potts (Chem. Phys. Res. Lab., The Dow Chemical Co., Midland, Mich., U.S.A.). *Appl. Spectroscopy*, 1960, **14** (4), 108–109.—The cell is constructed so as to be integral with a hypodermic syringe, so that a soln. may be drawn directly into the cell chamber. By inserting the needle into the solvent layer after extraction of aqueous solutions, the need for separation is avoided and transfer losses are minimised. The cell has been found most useful when extracting anaesthetics and common industrial solvents from blood samples.
P. T. BEALE

1328. **Sublimation of inorganic and addition compounds for infra-red spectroscopy.** H. A. Szymanski and P. Peller (Canisius College, Buffalo, N.Y., U.S.A.). *Appl. Spectroscopy*, 1960, **14** (4), 107.—The sample is placed in a platinum boat inside the copper coil of a 15-kW induction furnace. The boat can be heated to white heat in $\simeq 1$ sec., and the fine sublimate is collected on a rock-salt plate suspended above the furnace. Spectra of copper nitrate obtained by the new technique and from a liquid paraffin mull are shown, that by the sublimation technique being purer and more correct. Addition compounds such as the amine and dioxan complexes of the halides of As, Sb and Ti do not appear to decompose under the sublimation conditions.
P. T. BEALE

See also Abstracts—**896**, Automatic flame photometry and colorimetry. **898**, Proceedings of Colloquium Spectroscopicum Internationale, 1959: Spectrographic analysis by injection of soln. Uses of X-ray emission spectrography and β-ray fluorescence. **906**, Spectroscopically pure cyclohexane. **916**, Treatment of mass-spectrometric data. **1118**, The $\Delta\epsilon$ method of analysis.

Electrical

1329. **Radiometric titrations with radioactive silver-110.** P. Bebeşel and I. Sîrbu. *Rev. Chim., Bucharest*, 1960, **11** (5), 288–291.—A standard soln. of $^{110}AgNO_3$ is used. *Preparation of reagent*—Silver nitrate (1 g) is irradiated in a flux of 10^{12} *n* per sq. cm per min., dissolved in water and filtered. The soln. (**I**) ($\simeq 0{\cdot}1$M, of activity $\simeq 3000$ counts per min. per ml) is titrated with standard KIO_3 soln. (**II**). An aliquot of **I** (or **II**) is placed in a centrifuge tube, treated with a small known vol. of **II** (or **I**) and centrifuged. A portion of the clear supernatant liquid is withdrawn for measurement of its activity, then replaced, and the procedure is repeated so as to obtain a graph of counts per min. *vs.* vol. of titrant added, from which the end-point can be determined. The following determinations are described. (*i*) *Cyanides*—The test soln. is titrated with **I** as described above. The graph of activity *vs.* vol. of titrant at first rises, then falls, the end-point being the intersection of the two straight lines. The error for 0·3 to 4·0 mg of CN^- ranges from $-0{\cdot}12$ to $-0{\cdot}9\%$. (*ii*) *Oxalic acid*—The acid (after neutralisation with NaOH) and sol. oxalates can be similarly titrated with similar accuracy. (*iii*) *Sulphonamides*—**I** can be used for titrations by the method of Lee (*Analyst*, 1957, **82**, 185) without dichromate indicator; for 1·5 to 8·3 mg the errors range from $+0{\cdot}57$ to $-0{\cdot}8\%$. (*iv*) *Lead*—The test soln. at pH 7 is treated with a few drops of **I** (as indicator) and its activity is measured. Standard K_2CrO_4 soln. is added in small increments, and the activity is measured after each addition, the end-point being indicated by a sharp inflection in the curve. The error for 0·5 to 21 mg of Pb ranges from $+0{\cdot}15$ to $-1{\cdot}4\%$.
H. SHER

1330. **Liquid scintillation counting of aqueous samples.** B. A. Loveridge and A. M. Thomas (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report AERE-R2942, 1960. 28 pp.—The determination of β-activity is discussed with special reference to effluent and potable water. The equipment used is described and illustrated. The sample (2 ml) is mixed with a soln. (10 ml) of 2,5-diphenyloxazole (40 mg) and naphthalene (1 g) in dioxan (100 ml) and placed in a glass cell mounted between two photomultipliers connected in a coincidence circuit

to reduce the background count rate. The counting of α-particles, the effect of pH, and the quenching effects due to water and $Ca(NO_3)_2$ are briefly noted. The counting efficiency for β-particles of energy >0·06 MeV was ≮50% and for those of energy >0·12 MeV was ≮70%; it was 95% for particles with an energy of 1·0 MeV. G. J. HUNTER

1331. **All-Teflon counting cell for flowing radioactive solutions.** W. J. Blaedel and E. D. Olsen (Chem. Dept., Univ. Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1960, **32** (7), 789–791.—In the cell described, which can be used for the continuous monitoring of small radioactive effluent streams, the entrainment of air, hold-up, and mixing are minimised. Solution counting of β-emitters as weak as ^{45}Ca is possible. A few elements are strongly adsorbed by Teflon and the last traces cannot be removed even by extended washing. K. A. PROCTOR

1332. **Radiochemical analysis of multiply-labelled substances.** N. Getoff (Inst. f. Radium-forsch. u. Kernphys., Öst. Akad. Wiss., Wien). *Öst. Chem-Ztg*, 1960, **61** (4), 101–106.—A review is presented. The analysis of mixed radio-indicators requires the separate determination of the activities of the components, either after chemical separation of the nuclides or by the use of physical instruments that distinguish between radiations differing in kind or in energy. (35 references.) J. P. STERN

1333. **Absolute measures of β-activity. Application to the determination of the milligram radium equivalent.** S. Vuccino. *Ann. Phys.*, 1959, **4** (Sér. 13), 147–184.—A description is given of apparatus suitable for the measurement of weak (≃1 μC) and strong (>10 μC) radiation and for absolute measurements in an ionising chamber. Techniques and sample preparation for measurements by β-γ and γ-γ coincidences are described, and errors due to angular correlations and fortuitous coincidences are discussed. The determination is described of the "milligram Ra equiv." (*i.e.*, the amount of active substance, expressed in mC, that produces the same ionisation current in a given chamber as 1 mg of elementary Ra) of ^{64}Cu, ^{137}Cs, ^{46}Sc, ^{60}Co and ^{24}Na, using two types of condenser chamber. Results by the various methods are compared, and it is concluded that, for substances having simple disintegration patterns, methods based on coincidence measurements are satisfactory. The apparatus, once set up, can be used for serial measurements, no complicated preparation of samples is required, and the method permits the measurement of highly active samples. R. E. E.

1334. **New method of sample interchange in a solid-source mass spectrometer.** B. R. F. Kendall (Dept. Physics, Univ. Western Australia, Nedlands). *J. Sci. Instrum.*, 1960, **37** (4), 130–131.—Samples mounted at different positions along an electrically heated filament are at different potentials. There is thus a variation in (*i*) the efficiency with which ions are extracted from each sample and (*ii*) the kinetic energy of ions extracted. An ion source is described by means of which a number of samples so mounted can be analysed in a single loading and pumping cycle. G. SKIRROW

1335. **Introduction of analysed gas into the ion source of a mass spectrometer.** M. S. Chupakhin (V. I. Vernadsky Inst. of Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1960, **15** (2), 155–158.—The effect has been studied of the fractionation of isotopes when a glass diaphragm is placed in the channel of the feeding system of a mass spectrometer. The minimum gas vol. have been established for obtaining peak intensities which enable the variations in the isotope composition of natural gases to be recorded. The design of a three-channel feeding system is described, permitting the simultaneous comparison of two samples with a standard. A. BURWOOD-SMITH

1336. **Principle of a new, non-electronic integrator with figure registration.** L. Halász and W. Schneider (Scholven Chem. A.-G., Gelsenkirchen-Buer, Germany). *Z. anal. Chem.*, 1960, **175** (2), 94–96 (in German).—The evaluation of physical measurements for analytical purposes frequently necessitates the integration of a rapidly changing variable over a definite period of time, and the integrator described counts 1·8 million impulses per min. for the full scale width. A diagram of pulse emitter and diaphragm control is shown. The deviation of the integrator from linearity is $\not> \pm$ 0·2%, expressed as a percentage of full-scale deflection. B. B. BAUMINGER

1337. **Improvements in and relating to apparatus for continuously measuring the concentration of substances present in a fluid.** N. V. Algemeen Technisch Ontwerpbureau Alto. Brit. Pat. 840,935; date appl. 12.10.56. Netherlands, date appl. 14.10.55.—A bridge circuit for measuring the concn. of insol. electrically-insulating particles suspended in a liquid comprises cells for containing the reference liquid and the liquid to be measured, respectively. The arrangement is such that the impedance of the measuring cell, when filled with the same liquid as the reference cell, is equal to twice the impedance of the reference cell and that in this condition the bridge circuit is balanced. J. M. JACOBS

See also Abstract—**917**, Use of electron probe in analysis. **1058**, Applications of polarography in org. chemistry.

ERRATUM

November (1960) issue, abstract 5057, line 7. *For* "acetic anhydride" *read* "active carbon".

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.	milli-equivalent	milli-equiv.
ampere	amp.	milligram	mg
Ångström unit	Å	millilitre	ml
anhydrous	anhyd.	millimetre	mm
approximate, -ly	approx.	millimicrogram	$m\mu g$
aqueous	aq.	millimolar	mM
atmospher-e, -ic	atm.	millivolt	mV
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	M
calorie (large)	kg-cal.	molecul-e, -ar	mol.
calorie (small)	g-cal.	normal (concentration)	N
centimetre	cm	optical rotation	α^t_t
coefficient	coeff.	ounce	oz
Colour Index	C.I.	parts per million	p.p.m.
concentrated	conc.	per cent.	%
concentration	concn.	per cent. (vol. in vol.)	% (v/v)
constant	const.	per cent. (wt. in vol.)	% (w/v)
crystalline } crystallised }	cryst.	per cent. (wt. in wt.)	% (w/w)
cubic	cu.	potential difference	p.d.
current density	c.d.	precipitate (as a noun)	ppt.
cycles per second	c/s	precipitated	pptd.
density	ρ	precipitating	pptg.
density, relative	d or wt. per ml	precipitation	pptn.
dilute	dil.	preparation	prep.
direct current	d.c.	qualitative, -ly	qual.
distilled	dist.	quantitative, -ly	quant.
ethylenediaminetetra-acetic acid	EDTA	recrystallised	recryst.
electromotive force	e.m.f.	refractive index	n^t_λ
equivalent	equiv.	relative band speed	R_F
gram	g	relative humidity	r.h.
gram-molecule	mole	revolutions per minute	r.p.m.
half-wave potential	$E_{\frac{1}{2}}$	saponification value	sap. val.
hour	hr.	saturated calomel electrode	S.C.E.
hydrogen ion exponent	pH	second (time)	sec.
infra-red	i.r.	soluble	sol.
insoluble	insol.	solution	soln.
international unit	i.u.	specific gravity	sp. gr.
kilogram	kg	specific rotation	$[\alpha]^t_\lambda$
kilovolt	kV	square centimetre	sq. cm
kilowatt	kW	standard temp. and pressure	s.t.p.
liquid	liq.	temperature	temp.
maxim-um, -a	max.	ultra-violet	u.v.
melting-point	m.p.	vapour density	v.d.
microgram	μg (not γ)	vapour pressure	v.p.
microlitre	μl	volt	V
micromole	μmole	volume	vol.
micron	μ	watt	W
milliampere	mA	wavelength	λ
		weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc. for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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